

Experimental Laboratory Manual in Materials Science and Engineering

2nd Edition

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**Descriptors: / Industrial Engineering\ \
Mechanical Properties**

❖ أعدت دائرة المكتبة الوطنية بيانات الفهرسة والتصنيف الأولية.
❖ يتحمل المؤلف كامل المسؤولية القانونية عن محتوى مصنفه ولا يعبّر هذا المصنف عن
رأي دائرة المكتبة الوطنية أو أي جهة حكومية أخرى.

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1. Introduction:

The studying of materials and materials testing is not a new concept. People have been using materials since the beginning of time. Modern methods of study started with the development of the scientific method of study. Since then, numerous improvements have been made in testing.

Materials testing involves the identification of the properties of materials through well-established procedures. These procedures are conducted in a manner that is both accurate and precise. The significance of a test is in its ability to predict the performance of a materials in service.

Data collection refers to the procedures or operations that are performed to obtain data on a material or material specimen. The ability of the investigator to select the best possible procedure to follow, determines the effectiveness of the data collection. Even best procedures can fail if the investigator fails to follow directions properly, skips some of the procedures to save time, or records the information improperly.

Before beginning to conduct testing on materials, the researcher should ask the following questions:

- Where is this data going to be collected?
- When is the best time to collect the data?
- Who is going to collect the data?
- What data need to be collected?

Materials testing deals with the measurement or quantifying of values that indicate the properties of the materials. Mechanical properties are those that deal with the elastic or inelastic behavior of the material under load. The primary measurements involved in the mechanical testing are the load applied and the effects of load application. These measurements can be also called stress and strain, respectively.

The most common mechanical properties tests are tension, compression, impact, hardness, fatigue...etc. Ideally, a test should be purposeful, reliable, replicable, within a specified precision, and economical in both time and money.

The difference between the reading on the instrument and the true value of the measurement in question is the error. The measurement error is also a function of the instrument's sensitivity. The sensitivity of the instrument is its ability to detect and respond to a change in the characteristics being measured.

There are two general classes of error: methodical and coincidental. Methodical errors tend either constantly to inflate or constantly deflate the measured value. Coincidental errors are random occurrences that influence the results in a random fashion. They tend to cancel each other out. Coincidental errors cannot be corrected.

It is important to publish test results so that others may share in findings. Therefore, it is important that the report be clear in its meaning and form. A technical report of findings is necessary.

To help the students to practice materials testing in a systematic and scientific way, it was decided to collect our efforts in this book and present it to them as a guide.

This book is different from other books in its style, presentation, and in concentrating on the experimental part of the material science subjects with a short description of theory that helps student as a support or reminder to the classroom instructions in materials science with some hints to that subject in the textbook to which the student should refer. On the other hand, it contains lots of experiments such as macroscopic examination, metallographic preparation for microstructural examination, construction of phase diagrams, and surface hardening in addition to the most common mechanical tests such as hardness, fatigue...etc.

This manual is designed to supplement the classroom instructions in Materials Science and Engineering through demonstration of basic principles of material properties and heavily structured laboratory work to give the student a physical "feel" for some concepts in this subject. It was prepared to contain ten experiments that can help in translating the theoretical knowledge gained by the student to some thing that can be sensed. Appendices were attached to this manual which can help the students or researchers in different ways.

2. General Information

2.1. Class policy

- ♦ Reports shall be considered for full credit only if submitted upon, or before, the appointed due date. A penalty of 10% shall be assessed for each day that a report is late. Reports may be only submitted to the instructor or teaching assistant. Exceptions shall only be granted only upon medical grounds and are subject to the instructor's approval.
- ♦ Each report is due during the lab period following the completion of each experiment. Report preparation is an individual effort.
- ♦ Regular attendance is required. Excused absence shall be given only for a emergency or medical reasons and shall be subject to the instructor's approval.
- ♦ Ten quizzes shall be conducted before the experiment starts.
- ♦ Five reports and five quizzes of the highest marks are considered for the final mark.
- ♦ Refusal to submit reports shall be viewed as a severe academic irregularity. Negative weight shall be applied to the final grade for each report that is not submitted.

2.2. Safety Precautions

Safety in laboratory can be achieved only when common sense and consideration for others prevails, while following the basic regulations outlined below:

1. Smoking is not allowed in the laboratory.
2. All accidents should be immediately reported to the instructor.
3. Student should know the location of fire extinguishers and how to use them.
4. Experiments should never be left unattended.
5. Student should never taste any solid or liquid chemical.

6. If acids are spilled on your skin, wash with plenty cold water then consult your instructor.
7. It is advisable to wear safety glasses and gloves in the laboratory.
8. Always wear a laboratory coat.
9. If acid is spilled on your clothing, bench or floor wash thoroughly with water, then neutralize with dilute ammonium hydroxide or acetic acid respectively and inform your instructor.
10. Always wash your hands with soap and water on leaving the laboratory.
11. The student to be aware of the safety signs in order to avoid any undesired circumstances.

Safety sign is defined in the regulations as “a sign combining geometrical shape, color and pictorial symbol to provide specific health or safety information (whether or not any text is also included).”

Examples of the main types of signs are: Prohibition, warning, mandatory and safe conditions. Examples of these signs that comply with British Standard are shown below.

Prohibition (BS 5378 PART 1: 1980)



Fire (BS 5499 PART 1: 1984)



Fire hose reel

ISS F20B 200 x 150
ISS F20C 400 x 300



Fire extinguisher

ISS F16B 200 x 150
ISS F16C 400 x 300



Fire alarm

ISS F01C 200 x 150
ISS F01D 400 x 300



Fire phone

ISS F35B 200 x 150



ISS F26A
280 x 90



ISS F27A
280 x 90



ISS F28A
280 x 90



ISS F29A
280 x 90



ISS F30A
280 x 90

Danger - warning (BS 5378 PART 1: 1980)



DANGER
Explosive
material

ISS W03C 400 x 300



DANGER
Fork lift
trucks

ISS W10E 400 x 300



DANGER
Suspended
load

ISS W09A 400 x 300



DANGER
Falling objects

ISS W26B/R 400 x 300



DANGER
Overhead
hazard

ISS W13B 200 x 150



DANGER
Trip hazard

ISS W27B/R 600 x 450



DANGER
Slippery
surface

ISS W19B 400 x 300



DANGER
Overhead
live wires

ISS W14B 200 x 150



DANGER
Guard dogs

ISS W11B 400 x 300



DANGER
Corrosion
risk

ISS W05M 250 x 150
ISS W05N 400 x 300



DANGER
Scaffolding
incomplete

ISS W25B/R 600 x 450



DANGER
Toxic hazard

ISS W04K 200 x 150
ISS W04L 400 x 300



Danger
moving
machinery

ISS W36A 400 x 300



DANGER
Fire risk

ISS W02E 400 x 300
ISS W02H 200 x 150



DANGER
Highly flammable
material

ISS W08C 200 x 150
ISS W08D 400 x 300

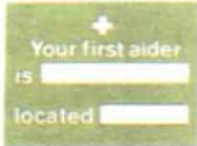
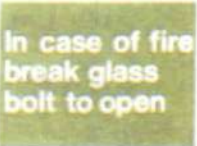
Mandatory (BS 5378 PART 1: 1980)



Safe conditions (Wy - Tags)



Safe conditions (BS 5378 PART 1: 1980)

 EXIT ISS E01F 150 x 450 (125)	 EMERGENCY EXIT ISS E02D 150 x 500 (50) ISS E02E 300 x 900 (100)	 FIRE EXIT ISS E03F 150 x 600 (100) ISS E03H 100 x 400
 FIRE EXIT KEEP CLEAR ISS E26A 225 x 600 (100/50)	 Assembly point ISS E10B 225 x 500 (50)	 FIRE EXIT ISS E03G 300 x 450 (125)
 Drinking water ISS E12A 75 x 50 ISS E12B 200 x 200	 First aid box ISS E27A 200 x 150 ISS E27B 67 x 50	 First aid stretcher ISS E31A 200 x 150
	 ISS E09E 150 x 40 ISS E09G 300 x 80	 ✓ Push bar to open ISS E14C 75 x 600 (50)
		 ✓ Push to open ISS E13B 50 x 175 (15)
 Emergency stop ISS E34A/S 75 x 50 ISS E34B 300 x 200 ISS E34C 200 x 150	 Emergency shower ISS E18B 350 x 200	 First aid ISS E04B 150 x 150 ISS E04C 300 x 300
		 eye wash ISS E22C 300 x 300
		 PUSH ISS E25A 150 x 37
		 PULL ISS E24A 150 x 37
 Slide to open ISS E10B 200 x 300 (40)	 Slide to open ISS E06B 200 x 300 (40)	 Your first aider is located ISS E42A 150 x 200
		 In case of fire break glass bolt to open ISS E11A 50 x 75 (5)
		 ISS E08E 150 x 150 ISS E08G 90 x 90 ISS E08F 100 x 100
INFORMATION TAPE 50mm x 33mm self-adhesive roll. To indicate safety services, medical stores, emergency showers, etc. ISS E24C Plasticised vinyl floor marking quality. ISS E24B Twin-ply laminated tape.		
		
	 ✓ Push bar to open ISS E14B 200 x 400 (50)	 ISS E09J 150 x 90 ISS E09K 90 x 90

If the symbol and/or text that you require is not illustrated on this page, please ask:

See separate price list and order form for prices, discounts and terms of business.

2.3. Presentation of data and findings

2.3.1. Technical report writing

Technical writing is characterized by its technical subject matter, impartial and objective presentation of facts, high concentration of complex definitions, descriptions and mathematical expressions, and extensive use of scientific graphic tools, including graphs and tables. As engineers, you will generally be required to present your work in the form of technical articles and reports. Published journal articles are much concise than reports because of the space limitations. Reports, in general, are self – contained and require very little outside reading for complete content understanding. For this stage, it will not be necessary to write the reports as if you invented and designed the laboratory experiments yourself. But the report should be written so that:

- ♦ Another engineer could understand your results and use the data without consulting with you.
- ♦ The Work you present to another person can play a large role in that person's impression of you and your ability (in neat and orderly fashion).
- ♦ All reports shall be typed and checked for spelling and grammatical errors.

A. Details

The following are some suggestions for general report writing. You will find, in subsequent courses and in industry, that these report characteristics are expected, not merely appreciated.

1. All reports shall be typed and checked for spelling and grammatical errors
2. All reports shall have a cover sheet, upon which the performance and due dates. the name of the experiment, the author, other group

members, and the course title shall all appear.

3. Organize your report, and clearly separate the different sections, using appropriate headings, numbered in accordance with their appearance in the contents page. (See the following report structure suggestions.)
4. The report shall have a contents page, with each of the sections numbered and listed by page number.
5. The first page of each section shall be numbered at the bottom center. All subsequent pages of each section shall be numbered in the upper right-hand corner. All pages within appendices shall be numbered with a capital letter (corresponding to the appendix name), a dash, and an Arabic number (i.e.--A-1, B-3, etc.). All of these page numbers shall be centered at the bottom of each page.
6. All figures and tables shall have a title and a number. The numbers shall be sequential. All figures and tables shall be referenced in the body of the report. The figures and tables may be either inserted into the running text or appended after the text, but before the appendices.
7. Figures shall be neatly drawn using straight edges, French curves, etc. Computer drawings are preferred. Avoid freehand sketching. See the Graphing section for details on producing acceptable curves.
8. Sometimes it may be necessary to use figures or data directly from others sources (or earlier experiments). Do not submit glued, taped, or stapled figures on the final report. If need be, photocopy the entire page. Of course, remember to acknowledge the source of such data.
9. Be concise and to the point. A technical report should not read like a novel. Always avoid using the first and second person (I, we, you, etc.).

The third person is appropriate.

10. Use the past tense at all times unless it's truly awkward.
11. A nice finishing touch is to enclose the report in a cover. A variety of inexpensive covers are available at most bookstores. Professional bindings are also acceptable and preferred if the report's volume exceeds the capacity of slip-on covers (usually about 25 pages).

B. Form of the Report

The format described here is based upon that which is commonly used in various research organizations. It should serve as an organizational framework for your reports. The major divisions of your report shall be:

Introductory page:

0.0 *Title page. It includes the name of the university, the name of the faculty, the name of the department, the title of the experiment, the name of the student, the name of the supervisor.*

Body of the report:

- 1.0 *Introduction*
- 2.0 *Review of Basic Theory*
- 3.0 *Materials, Equipment, and Experimental procedure*
- 4.0 *Results*
- 5.0 *Discussion*
- 6.0 *Conclusion*
- References*
- Bibliography*
- Appendices*

A numbering scheme, similar to that above, shall be used for the section titles of each

report and the content of each of the major mentioned above divisions can be illustrated as follows:

1.0 Introduction

The introduction serves to orient a competent technical reader to the particular subject matter of your report. It should include general statements familiarizing the reader with the problem under study, the laboratory technique used, and the experimental objective to be obtained. The purpose of the experiment should be clearly outlined in this section. It should be clear from this section why the experiment was needed in the first place and what the outcome of such an effort will be.

A poor introduction:

In this experiment, the hot plate was positioned in a vertical plane. Pictures taken near thermocouples 11 and 13 determined the air densities there. These numbers were used to compute temperatures in the moving air.

A proper introduction:

The thermal energy dissipated by a vertical hot plate induces a free-convective laminar flow in the surrounding air. The temperature distribution within this laminar flow layer may be determined from air density measurement, obtained by the method of optical interferometry. The purpose of this experiment is to compare the temperature distribution determined, through the use of a Mach-Zehnder interferometer, to the temperature distribution predicted by theory.

2.0 Review of Basic Theory

Summarize the important theoretical assumptions and present the governing equations with some explanation of their origins (a derivation may be appropriate in some cases). Theorems which are central to the purpose of the experiment or to the final

evaluation of the data are to be presented here. For example, if the experiment concerned the determination of the principle stresses of a structure with combined loading, present the definitions which are essential. Derivations of these theorems are not necessary. Rather, their relevance to the experiment should be explained. Present these theorems in their mathematical forms and define all symbols used. It is sometimes customary to include the definitions of all symbols either at the very beginning of the report (before the Introduction) under the heading Nomenclature or at the very end of the report under the heading Notation.

Experiments that involve no theoretical analysis do not require the presence of this section in the body of the report.

3.0 Materials, Equipment, and Experimental procedure

This section should focus on mentioning the materials used in the experiment and their characteristics, the main equipment necessary to conduct the experiment, and the important details of the physical set-up of the experiment and the experimental procedure that was followed. A schematic diagram of the laboratory apparatus should be included or referenced in this section. Remember that the reader is familiar with your project. Describe any particularly important details, such as how and under what conditions the data was collected. Describe what was used as criteria to determine that the data was ready to be recorded. Especially note any changes or adjustments which were necessary for satisfactory completion of the experiment. Remember that other similar projects may be performed based upon your written report. Make adequate record of the particular equipment used. This can range from a list of all equipment, including serial numbers, to a simple notation of which experimental set-up was used.

4.0 Results

The result of an experiment is the reduced data presented in the form of graphs, charts, or tables. In general, the reduction of data from the measured raw data requires some calculation. If some additional theoretical background is necessary for such a reduction, it should be discussed in the Basic Theory section. A sample calculation of the reduction of data should be included either in this section or in an appendix.

5.0 Discussion

Discuss the results in light of the basic theory. Any explanations of particularly good or bad results should be placed here. Explanations of any strengths and weaknesses of the experimental and computational procedures are also appropriately placed here. The questions posed in the lab handout also belong here.

6.0 Conclusion

A brief summary of the important results and a brief emphasis of the important points of the discussion belong here. Comments with regard to the general purpose of the lab and suggestions as to possible applications of the results or further investigations should be included here. These are your individual comments and should reflect any original thoughts or additional results that were obtained.

References

The literature, to which direct reference was made in the report, shall be placed here. Paraphrasing and direct quotes are to be noted by raised numbers in the report. Placement of the literary references shall be in the sequential order of their

Bibliography

It is very important to give proper credit to the references used in the report. This section also shows the extent and depth of research done before the report was written. The bibliography listings shall be alphabetical in placement and shall follow

any of the currently accepted styles being taught in today's English classes. These listings shall include, but are not limited to, all of the works cited in the References section.

Appendices

All detailed calculations, tables of laboratory data, analog and/or digital results, data sheets, strip charts, computer programs, etc. should be inserted in suitable appendices at the end of your report. These items are important, however, when included in the main body of the report, such material constitutes a confusing digression from the main line of argument. Thus, it is best to relegate this material to an appendix, where an interested reader may access it if he so chooses. Organize and label the appendices so that the reader may quickly find any particular data or calculation set.

Laboratory data sheets shall contain data that is expressed to the same degree of accuracy as was provided by the instrument read-outs. Computed data shall also have its accuracy restricted by the experimental data from which it was found.

C. Presentations of the Graph

Most reports shall require one or more curves. These curves summarize the data and the results. The reader is often able to visualize trends and results that are not readily apparent from written data. Curves are therefore very important and should be constructed as carefully as possible. The following specifications apply to all graphs and curves:

a. Paper:

If drawn with the aid of straight edges and French curves, graphs shall be drawn on 20 x 20 lines-to-the-inch coordinate paper, or some other suitable scale. Standard white, typing paper is acceptable for computer draw curves.

b. Axes:

1. The intersection of axes and their scales should be selected so that a maximum area of the coordinate paper will be utilized. Scaling should make efficient use of the tic marks and ruled lines already on the page. Computer generated curves should also have their areas used in the most efficient manner.
2. The axes shall be labeled with a descriptive title (not just "abscissa vs. ordinate"). This title shall include information specific to the data set (i.e.--which cases were run, which materials were tested, or other brief information).
3. Each axis shall be labeled with the appropriate units of measure, including any powers of ten by which they are multiplied.

c. Plotted points:

1. All observed values shall be plotted as fine, clean, tiny dots.
2. A circle, about 0.1 inch in size, shall be drawn around each point.
4. When more than one curve is place upon the same page, symbols other than the circle should be used for each different curve. Permissible symbols, in the order of their suitability, are the square, triangle, diamond, and cross.

d. Smooth curves:

1. A smooth curve shall be drawn to represent the average of the plotted points. It should be a fine but distinct line, drawn with a French curve and/or straight edge. Each plotted point represents experimental data and the curve is the student's interpretation of the entire data set.
2. Where there appears to be a lack of definite relationship between the

plotted values, a point-to-point curve or series of straight lines may be drawn. The curve shall not pass through the circles enclosing the plotted points it should stop at their circumferences.

3. Supplementary lines (i.e.--yield strength at 0.2% offset, etc.) are used, they shall be drawn lightly or dashed and shall not be erased.
4. All points of interest on the curve shall be labeled clearly.

e. Title:

1. A descriptive title, not just "abscissa vs. ordinate" shall be used. Include the cases that were run, material used, the loading applied, the type of curve (stress-strain, torque-twist, toad-deflection, etc.), or specimen description. Whatever is most appropriate.
2. The preferred location for the title is centered above the axis, slightly higher than the tip of the y-axis. A secondary location is the lower, right-hand corner of the page.

f. Legends:

A legend, or key, that notes all symbols shall be placed upon all diagrams containing multiple data sets.

D. Curve Correction

Oftentimes the instruments used to measure some phenomenon do not register any readings until a considerable load has been applied to the specimen. This "gap" is produced by the instrument's necessity to "take up slack." This "slack gap" results in the curve beginning some distance from the origin. Since it is the purpose of all experiments to get accurate data, free from errors in instrument readings, a correction of the data is necessary.

Such a correction of a "slack gap" is easily performed upon stress-strain curves by

making use of certain of their characteristics. A stress-strain curve should obviously pass through the origin of the coordinate axes (since there is no strain in an object if there is no stress applied). The load (or stress) is determined directly (or indirectly) from readings taken from the weighing apparatus of the testing machine. If the machine was balanced before testing began, then the load readings should be accurate. The readings along the vertical axis (upon which the load is usually plotted) of the uncorrected graph are, therefore, usually correct. It is along the horizontal axis (upon which the strain is usually plotted) that errors will exist if the uncorrected curve does not pass through the origin.

To correct such a curve it is necessary to merely extend the straight line portion of the curve through the vertical axis until it intersects an extension of the horizontal axis. The point of intersection is then the true origin and the correct vertical axis may be passed through the intersection by simply sliding it to the left. No change in the calibration of the vertical scale will be necessary. Since the origin has moved along the horizontal axis, however, the calibration of the horizontal scale must be changed. An addition should be made, to each designated value on the horizontal axis, of an amount equal to the value of the scaled distance through which the vertical axis was moved.

Performing this correction forces the curve to pass through the origin, not by moving the curve, but by moving the origin. All properties derived from a curve should be taken from the corrected curve. If no portion of the straight-line part of the stress-strain curve exists on the uncorrected curve, the correction may not be made by using the method outlined above.

Note: Computer generated curves are preferred. For those students without access to a drafting package, the following manual instructions are provided.

F. Common Mistakes

Common mistakes can be classified as follows:

a. Improper arrangement of the report:

1. All sections of the report should follow each other in numerical order (i.e.--1.0, 2.0, 3.0, etc.).
2. A typed, organized form of the data sheet shall be placed within the Results section. The original shall appear in an appropriate Appendix.

b. Failure to number pages properly:

1. The first page of each section shall be numbered at the bottom center.
2. All subsequent pages of each section shall be numbered in the upper right-hand corner.
3. All pages with in the Appendices shall be numbered with a capital letter (corresponding to the Appendix name), a dash, and an Arabic number (i.e.--A-1, B-2, etc.). This number shall be placed at the bottom center.

c. Failure to title and/or label graphs:

See Section IV, Graphing, for the proper form and placement of the title and labels.

d. Poor graph construction:

1. The graph is the most important part of each report.
2. See Section IV, Graphing, for the proper form for each graph.

e. Poor conclusions:

1. See the Conclusion paragraph of the Form of the Report section

for a description of what is desired in a conclusion.

2. Where comparison of experimental results, to accepted values is required, the student should list the accepted numerical values. A simple statement the experimental

results "compare favorably with accepted values" is not sufficient.

3. A simple numerical comparison of the experimental and accepted results does not comprise a complete discussion of the results. Refer to the objective of the experiment and make a conclusion as to whether or not the objective was accomplished.

f. Poor grammar and/or spelling:

1. All reports shall be proofread before they are submitted. Ask a classmate to proof your report after you have done it yourself.
2. Grammar shall be consistent with current English standards and should get the author's point across simply and with a minimum of wording.
3. All spelling shall be correct. Computer generated reports may be spell checked through whatever software dictionary the student has available.
(Note: This is not proofreading. Most spell-checkers will not catch a correctly spelled word placed incorrectly.)

4. Commonly misspelled words:

- i. SPECIMEN (not speciman),
- ii. SPECIMENS (plural; not specimen or specimans),
- iii. RIVET (not rivit),
- iv. FLEXURE (not flecture),
- v. SHEAR (not sheer),
- vi. TENSION (not tention), etc.

3. EXPERIMENTAL UNCERTAINTY

3.1 Introduction

When one is interested in learning about a physical problem, an approach, which provide considerable insight is an investigation involving measurement of properties of the phenomena. This approach is particularly useful when it is not possible to gain understanding by solving the questions (if available) which represents the physics of the problem.

While attempting to learn about a given physical property experimentally, rarely does an experimentalist determine the exact quantity in completely direct manner. Rather, the physical quantity of interest is usually determined through a measurement chain consisting of the following parts:

1. Probe
2. Transducers.
3. Manipulation element.
4. Recording or display element.

In order for the measurement to be meaningful, the experimentalist must then understand the relations between the different parts of the measurement chain as well as the relation between the physical quantity

To be measured and the measurement chain. Only then, it is possible to determine the relation between a final displayed output and the quantity of interest.

The parts of the measurement chain, may be further described as follows:

1. The **probe** is the device which detects and converts the physical Quantity of interest into some form of signal detectable by transducer.

In doing this, the probe should provide a clear relationship between the transducer and the quantity of interest without disturbing the phenomena, which is detected.

1. The **transducer** transforms the physical quantity of interest into a form (usually electrical) which can be more convenient for interpretation.
2. The **manipulation element** is a device used for acquisition and transmission of information. The manipulating element receives the output from the transducer and transmits it throughout the measuring system with minimal alteration.
3. The **recording and/or display element** transforms the output from the manipulation element to a permanent or semipermanent form, which can be readily interpreted. The interpretation may then be direct, or may require further data reduction and the use of computer.

In addition to understanding this measurement chain, the experimentalist must have some knowledge of the physical process of the environment to be measured. Knowledge of the behavior of the system tells which physical quantities should be measured. Also, knowledge of the characteristics of the system tells how these should be measured. For example, without a knowledge of the system, one cannot tell where or how to place the probes in an environment.

Knowledge of the effect of the measurement probe on the physical environment to be measured is also necessary. The probe used for measurements should provide as minimal disturbance to the environment as possible.

If the measuring environment is greatly altered by the presence of the probe, then the measurement using the probe is less meaningful. Probes should be designed for a given application so that maximum information at minimum disturbance is obtained.

Some useful definitions for measurement techniques are defined as follows:

Static characteristics: characteristics of the system, which are independent of the time.

Static error: true value-reading of instrument.

Accuracy: closeness with which a reading approaches the true value.

Intrinsic or ideal accuracy: accuracy achieved when calibrated laboratory conditions as opposed to working conditions .

Point accuracy: accuracy stated as percentage at one or more points on a scale.

Accuracy may be expressed as percent of full-scale range, which results in the occurrence of large errors at the low end of a scale. A more useful concept may be the expression of accuracy as percent of the true value.

Range: from "a" to "b", where "a" and "b" represent the limits of instrument use.

Span: : "a" minus "b".

Sensitivity: (1) magnitude or percentage change in the reading produced by a unit change in the measured variable, or (2) the smallest change in the variable which produces a noticeable change in output.

Reproducibility: degree of closeness with which a given value maybe repeatedly measured; the absence of drift (slow changes with time) means high reproducibility; units/given period of time.

Dynamic characteristics: characteristics of a system, which are time dependent, the dynamic characteristics of an instrument are usually determined by subjecting it to known variations in the measured quantity: step, linear, or sinusoidal.

Speed of response: the rapidity with which an instrument responds to changes in the measured quantity.

Lag: delay in the response of an instrument.

Fidelity: the degree to which an instrument indicates the changes in the measured without dynamic error.

Dynamic error: the difference between the true value of a time varying quantity and the indicated value.

Drift: the shift of an instrument calibration with time. No drift means perfect reproducibility.

Zero drift: shift of instrument calibration caused by a permanent set of slippage.

Span drift: a calibration change by a proportional amount caused by a gradual change in measurement characteristics.

Dead zone: range of values for a measured variable, the one that the instrument doesn't regard.

Experimental uncertainty: the range of values about center or mean value in which a given percentage of measurements are expected to occur.

Experimental operating domain: the limits or range of operation of experimental apparatus determined from power requirements and other variables of operation.

3.2. Uncertainty analysis and experimental error

Knowing the experimental of a measurement allows comparison to be made with the same measurement taken under different circumstances for example, two experiments conducted to measure the same quantity using the same equipment but at different facilities by different persons would not be expected to produce the exact same results. Rather, differences between the results would be expected resulting from the instrument calibration differences, human factors influencing the measurement and other effects. The results from experiments would be expected to fall within an interval about each actual measurement point. These intervals or bands may be referred to as experimental uncertainty, and each experiment should then be within the uncertainty interval of the complimentary experiment. The experimental uncertainty takes into

account the error incurred within a given measurement. According to Abernethy and Thompson there were two types of errors: fixed, and random errors. Random errors can be seen in the repeated trials of an experiment conducted using the same equipment by the same personnel. As repeated trials are made, slightly different values of data points are obtained due to numerous small effects. In other words the data scatters around mean value which can be estimated accurately if enough data points are taken. The standard deviation of the data about the mean value is then a measure of this random or precision error. A large standard deviation means a lot of scatter of measurements. The random error for a given measurement is illustrated in figure 1. The second type of error is fixed error, which may be also referred to as the bias, the constant error, or the systematic error. In a given experiment the bias is the difference between the mean value of the measurements and the actual value of the quantity being measured. The bias would be expected to be constant for repeated trials of the same experiment but may vary if the experiment is conducted by different personnel at a different facility. The bias is illustrated in figure 2 and may be difficult to calculate since the true value of the quantity being measured may not be known. In such situation, an upper bound on the bias must be determined by engineering judgment.

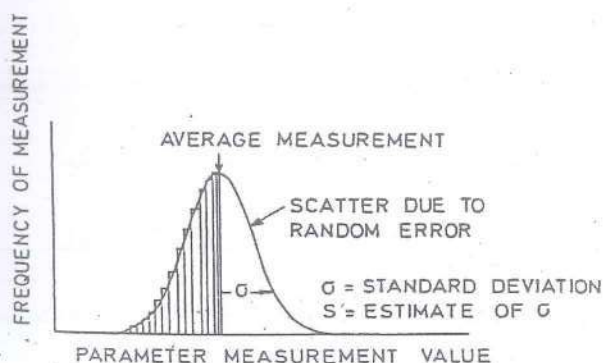


Figure 1. Precision error

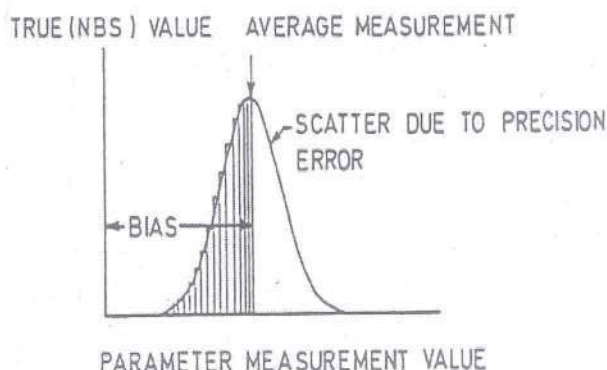


Figure 2. Bias error

The uncertainty value quoted for a given measurement may give the data measurement interval for random error, only. Such a calculation correspond to what Moffat refers to as "first order replication" uncertainty. In such a case, the uncertainty interval would reflect only those aspects which are not "fixed" during repeated trials. "Uncertainty" The calibration of the instrument shouldn't appear, because that aspect of the system would remain in variant during repeated trials. Alternatively an uncertainty may give the total measurement interval for a given measurement regardless of whether the data point is repeated using different equipment, different personnel, or in a different laboratory. Moffat calls this "Nth order replication" uncertainty.

In specifying an uncertainty it is important to realize that a value of an uncertainty is always dependent on the odds that another data point will fall into specified data interval. Odds are often referred to as the confidence level for the measurement uncertainty. For a given uncertainty interval the confidence level should increase as the experimental measurements are made more carefully. Also, the smaller the uncertainty bond for a given measurement, the looser the odds. In other words, fewer measurements are expected to fall within a given interval, as the interval decreases in size. The experimental uncertainty may then be specified as shown in equation (1.1).

$$R = 90.1 \quad \pm \quad 0.1 \quad (20:1) \quad (1.1)$$

The observed value	Uncertainty	The confidence level
--------------------	-------------	----------------------

Equation (1.1) then states that 19 out of every 20 measurements are expected to be equal to or between the value 90.0 and 90.2. the 2:1 odds confidence level corresponds to plus or minus two standard deviations about the mean if the scatter in the measurement points follow a Gaussian or Normal distribution.

Experimental uncertainty depends not only on the raw data from an experiment, but also on the operations involved to calculate the desired output from this data. Some calculations will amplify the uncertainty and some calculations will suppress the uncertainty.

Consider a result, R , calculated from a set of independent measurements, the x_i affected by various other aspects of the experiments such as instrument calibrations, test procedures, or hardware design. Denoting these aspects of the experiment as P_j , R can be expressed in terms of x_i and P_j as shown by equation (1.2).

$$R = R(x_1, x_2, x_3, \dots, x_n; p_1, p_2, \dots, p_m) \quad (1.2)$$

Result variables parameters

The computing equation, R , is the data reduction program. The values of x_i are the inputs required. The result, R , is the numerical value of the output. The parameters don't explicitly appear: their effect is "behind the scenes".

The experimental uncertainty associated with R , designated ∂R , can then be considered depending on the order of replication of R , according to Moffat. The zeroth order replication fixes time and all other parameters, such that:

$$\delta R_0 = \delta R (\delta x_1, \delta x_2, \delta x_3, \dots, \delta x_n) \quad (1.3)$$

first order replication permits time t vary and indicates the uncertainties due to nonsteady behavior, $\delta \theta$. Thus,

$$\delta R_1 = \delta R (\delta x_1, \delta x_2, \delta x_3, \dots, \delta x_n, \delta \theta) \quad (1.4)$$

If an investigator is to compare his work with that of other investigators, an Nth order replication level must be considered. The Nth order replication permits time to vary, includes the uncertainties due to non-steady behavior, and considers instrument identity as possible random variable. δR_n is then given by:

$$\delta R_n = \delta R (\delta x_1, \delta x_2, \delta x_3, \dots, \delta x_n, \delta \theta, \delta p_1, \delta p_2, \delta p_3, \delta p_n) \quad (1.5)$$

δR may be calculated one of several ways, if each x_i were simultaneously perturbed by an amount dx_i , then δR can be expressed using equation (1.6).

$$\delta R = (\delta R / \delta x_1) dx_1 + (\delta R / \delta x_2) dx_2 + \dots + (\delta R / \delta x_n) dx_n \quad (1.6)$$

Equation (1.6). Gives an improper estimate of δR when some x_i are positive, and others are negative. This is because δR is reduced to a net value not as large as it might be. One way to combine individual effects and preserve the true statistical probability is given by Kline and McKlintock. Using their method, δR is given by equation (1.7). Below:

$$\delta R = \{ [(\delta R / \delta x_1) dx_1]^2 + [(\delta R / \delta x_2) dx_2]^2 + \dots + [(\delta R / \delta x_n) dx_n]^2 \}^{1/2} \quad (1.7)$$

Where (1) each variable in (1.7). Is independent and comes from a Gaussian distribution, and (2) each variable in (1.7). Described by a statement of the form:

$$X_i = x_i \pm \delta x_i \text{ at (odds)} \quad (1.8)$$

Where the same odds are used for each of the variables. Alternatively, the percent uncertainty of R maybe determined by examining the logarithmic derivative of the equation relating the uniform quantity to measured variables. A maximum possible error maybe then computed by taking the root mean square of the error terms.

Two different measurements are considered to show agreement when the uncertainty bands of the two data points overlap. The agreement is considered good between two different measurements when one of the mean values for the data is contained within the uncertainty interval of the compared measurement point. (Uncertainty interval = $\pm$ two standard deviations for a 95% confidence level, as 20:1 odds). The two measurements are considered to be in excellent agreement when the data points lie within one half of the band for good agreement. This corresponds to an uncertainty level of plus, minus one standard deviation for a 95% confidence level.

The experimental uncertainty for the Nth order replication of a given measurement takes into account both the bias and the random error. An approach different from that previously discussed may also be used for calculation of uncertainty, dR . This method is suggested by the United States national bureau of standards, and is also used commonly in American industry. The method calculates the experimental uncertainty as the bias limit plus a multiple of the "precision error index" as shown in figure (1.3). When the uncertainty is centered about the measurement.

$$\delta R = \pm (B + t_{95} S) \quad (1.9)$$

When the uncertainty is not centered about the measurement, the upper limit of the uncertainty is the sum of $t_{95} S$ and the upper limit of the bias interval, B^+ . The lower limit of the uncertainty is defined by the lower limit of the bias interval, B^- , added to S . The uncertainty interval t_{95} Then ranges from dR^- to dR^+ , where

$$\delta R^+ = B^+ + t_{95} S \quad (1-10)$$

And

$$\delta R^- = B^- - t_{95} S \quad (1-10)$$

The experimental uncertainty interval for a non-symmetrical bias limit is illustrated in figure (4).

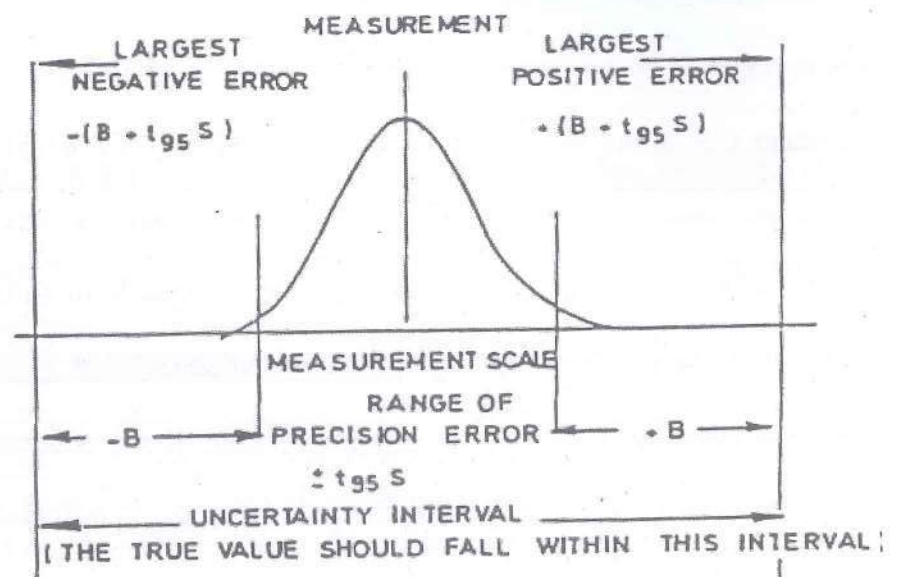


Figure 3: Measurement uncertainty, symmetrical bias

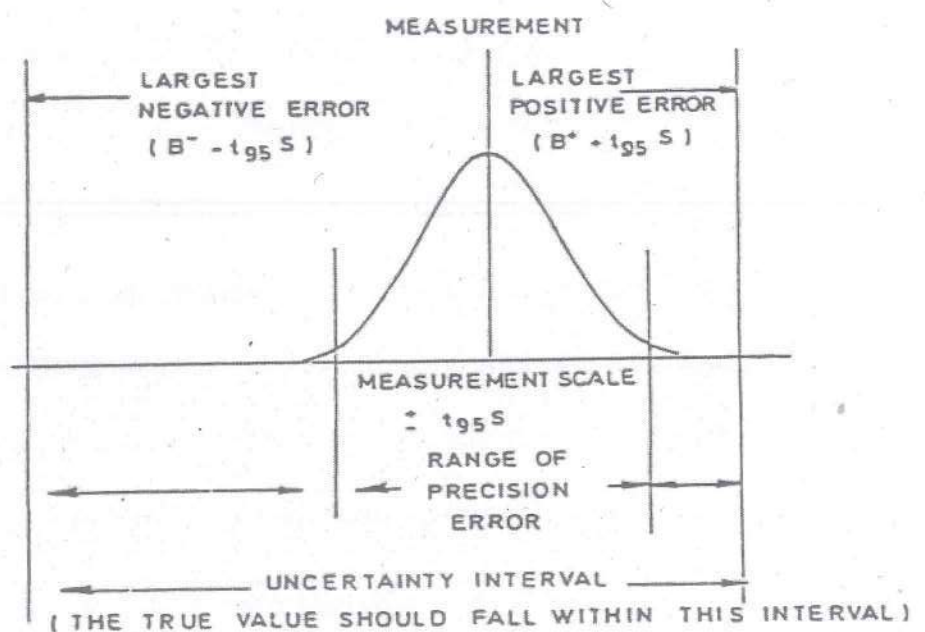


Figure 4: Measurement uncertainty, non-symmetrical bias

4. EXPERIMENTS

**SECTION I. DETERMINATION OF GENERAL
PROPERTIES OF MATERIALS AND MACRO AND
MICROSTRUCTURE**

EXPERIMENT No.1

Properties of Ferrous and Non-Ferrous Metals and Alloys

I. Purpose

Metals are designated as either ferrous or nonferrous. Today many hundreds of ferrous and nonferrous metals are in use. In general, nonferrous metals are more costly than ferrous metals. It is not always easy to distinguish a nonferrous from a ferrous metal, nor to separate one from another. This experiment introduce you to several methods used to identify and compare properties, select and properly use many of these materials.

II. Background

Review the sections in your materials science textbook covering the subject : types of materials.

III. Introduction

There are several methods of shop testing that you can use to identify or compare properties of ferrous and nonferrous metals and alloys. The testing methods are given to identify ferrous metals based on physical, chemical, and other properties of the material.

The following are some of the methods of shop testing that you can use to identify or evaluate some properties of ferrous, non ferrous, and metals and non metals alloys.

Visual Observation test

Some metals can be identified by visual observation of their finishes. Heat scale or black mill scale is found on all hot-rolled (HR) steels, these can be either low carbon

(0.05 to 0.30percent), medium carbon(0.30 to 0.60 percent), or high carbon (0.60 to 1.75 percent),or alloy steels (Figure 1). Other surface coatings that might be detected are the sherardized, plated, case-hardened, or nitrided surfaces. Sherardizing is a process in which zinc vapor is inoculated into the surface of iron or steel. Figure 2 reveals some of these textures.

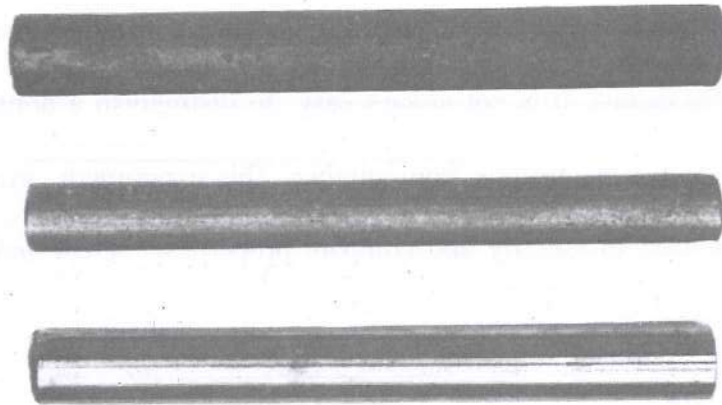


Figure 1. Rolled bars: top-hot rolled, centre-cold rolled, and bottom-ground and polished

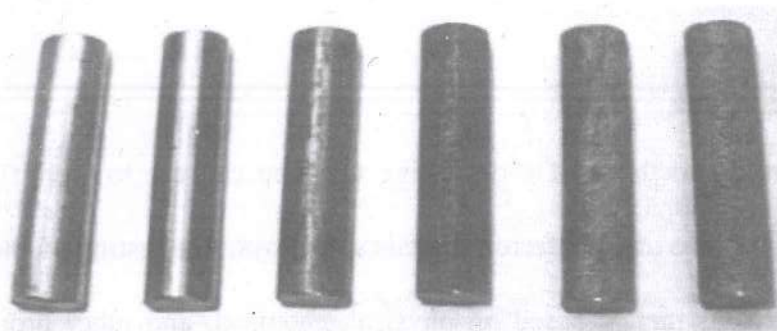


Figure 2. Round bars having various surface finishes. Left to right: aluminum, ground and polished, cold finished steel, hot-rolled steel, sheradized surface, and zinc dip surface.

Cold-finish (CF) steel usually has a metallic luster. Ground and polished (G and P) steel has a bright, shiny finish with closer dimensional tolerances than CF. Also cold-

drawn ebonized, or black finish are sometimes found on alloy and resulfurized shafting.

Chromium nickel stainless steel, which is austenitic and nonmagnetic usually has a white appearance. Ferritic steel is magnetic with a bluish-white color. Manganese steel is blue when polished but copper colored when oxidized. White cast iron fractures will appear silvery or white. Gray cast iron fractures appear dark gray and will smear a finger with a gray graphite smudge when touched.

Magnet Test

Most ferrous metals such as iron and steel are magnetic (that is , they are attracted to a magnet) but most nonferrous metals are nonmagnetic except nickel, which is nonferrous but magnetic. Since U.S. "nickel" coins contain about 25 percent nickel and 75 percent copper , they do not respond to the magnet test ,but Canadian "nickel" coins are attracted to a magnet since they contain more nickel . Ferritic and martensitic (400 series) stainless steels are also attracted to a magnet and thus cannot be separated from other steels by this method (See Chemical Tests below). Austenitic (300 series) stainless steel is not magnetic unless it is work hardened.

Density & Specific Gravity

(Density is absolute weight per volume ; specific gravity is relative weight)

The specific gravity of a symmetric or regular shaped solid may be found by measuring the piece to determine its volume and then weighing it on a scale. The density or mass per unit volume thus obtained may then be compared to Table1 in appendix D , specific Gravity (Density) and Weights of some materials. Thus, it is possible to identify the metal if it is a pure metal and if the measurements are

accurate. The volume and mass of an irregularly shaped solid may be determined by its displacement of a liquid of known density. This experiment is usually carried out in a chemistry or physics laboratory , using special equipment.

Selection for uses

There are a great variety of carbon, alloy, and tool steels to select for a certain application. Several types of cast irons are also available to the welder, machinist, or machine designer. The properties required for that certain application is the basis for choosing this or that material. Below is shown how some of these properties can be checked.

File Tests

Files can be used to establish the relative hardness between two samples. As in the scratch test, the approximate hardness of a piece of steel can be determined. *Table 1* gives the Rockwell and Brinell hardness numbers of some files. This method depends on the skill of the user. Care must be taken not to damage the file, since filing on hard materials may ruin the file. Testing should be done on the tip end or on the edge of the file (Figure 3).

Table 1. File test and hardness (Callister 2009)

<i>Type of steel</i>	<i>Rockwell B</i>	<i>Rockwell C</i>	<i>Brinell</i>	File Reaction
Mild steel	65		100	File bites easily into metal. Machines well but makes built up edge on tool.
Medium carbon steel		16	212	File bites into metal with pressure. Easily machine with high speed tools.
High alloy steel		31	294	File does not bite into metal except with difficulty.
Tool steel		42	390	Metal can only be filed with extreme pressure. Difficult to machine.
Hardened tool steel		50	481	File will mark metal but metal is nearly as hard as the file. Machining is impractical.
Case hardened parts		64		Metal is as hard as the file : should be ground.



Figure 3. File testing for hardness. (Lane Community College)

Chemical Tests

Many shops may have the ability to conduct simple chemical tests. Complex chemical tests should be done by a chemist. The surface to be tested must be very clean and free of all scale or oil. By applying only one drop of the acid with an eye dropper, a solution of 6 percent nitric acid in methanol (wood alcohol) will etch, or darken, carbon steel but will not discolor stainless steels (Figure 4). A 10 percent nitric acid solution will etch mild steel almost immediately, while the 6 percent solution takes about one minute. A drop of concentrated copper sulfate will leave a copper-colored spot on clean iron or steel but not on austenitic stainless steel. Some stainless steels react with sulfuric and hydrochloric acids. Types 302 and 304 of stainless steel possess a high affinity to sulfuric acid. Types 316 and 317 are attacked more slowly, leaving a tan surface.

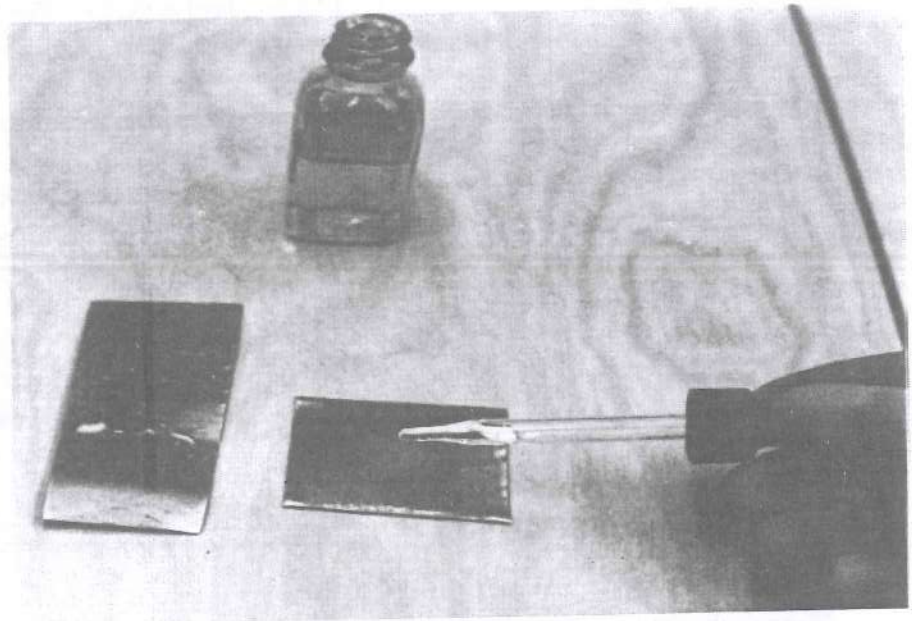


Figure 4. Identifying stainless steel with an acid test. A 6% of nitric acid in methanol (nital) has been applied to the stainless steel on the left and is being applied to the mild steel on the right. The mild steel is discolored, but the stainless steel is not.

Hydrochloric acid (HCL) reacts with types 304, 321 and 347 very rapidly, releasing gas. Type 302 leaves a pale blue-green solution on the surface, while types 303, 414, and 430F have a spoiled egg odor and leave a heavy black smudge. Steels containing selenium emit a garlic odor when attacked by HCL.

Commercial spot testers are available in kits. Stainless steel and many ferrous and non ferrous alloys can be readily identified using these test kits. Table 2 shows the chemical tests and reactions for some metals.

Table 2. Chemical tests and reactions for some common metals (Neely, 1989)

Reagents	Carbon steel	Stainless Steels				Aluminum	Magnesium	Nickel	Monel	Inconel
		Types 302, 304	Types 316, 317	Types 304, 321, 347	Types 303, 414, 430F					
Nital — 10% nitric acid in methanol	Blackens surface	No reaction					Cleans surface or turns gray	Slow reaction, turns blue-green	*Slow reaction, turns blue-green	No reaction
Hydrochloric acid	Reacts to clean surface	No reaction		Attacks, releasing gas	Attacks, leaving black smudge, rotten egg odor	Blackens surface	Blackens surface	Cleans surface	Cleans surface	No reaction
Copper sulfate solution	Leaves copper color	No reaction					Reacts, leaving heavy black smudge	No reaction		
Sodium hydroxide	No reaction					Reacts, leaves gray smudge	No reaction			
Sulfuric acid	Cleans surface	Leaves dark surface with green crystals	Slow attack, leaving tan surface	No reaction			Strongly reacts, foams, and leaves clean surface	No reaction		
Zinc chloride solution in water	No reaction						Reacts and turns black	No reaction		
Ferrous chloride	No reaction						Reacts and turns black	No reaction		

*Note. Monel may be distinguished from nickel (they look alike) by the following test. Place a few drops of nital on the surface of the specimen. It will turn blue-green if it is either monel or nickel. Then place a small piece of soft iron (a nail will do) in the solution on the sample. If there is no reaction, the metal is nickel, but if the iron turns copper colored, copper ions have been transferred from the copper in the metal, and it can be assumed that it is monel, which contains copper. Zinc and tin will react with so many acids to turn them black that chemical tests would be of no value for them. Silver will turn black in sulfuric acid. However, most other metals may be identified by testing for ferro-magnetism, density, color, or thermal conductivity.

Hardness Test

The hardness of a metal is its ability to resist local plastic deformation. There are many ways of hardness measurements: resistance to penetration, elastic hardness, and

resistance to abrasion are some of these ways. Here, the hardness of metals will be determined by their resistance to penetration.

Hardness varies considerably from material to material. This variation can be illustrated by making an indentation in a soft metals such as aluminum and then in a hard metal such as alloy tool steel. The indentation could be made with an ordinary center punch and a hammer, giving a light blow of equal force on each of the two specimens (Fig.5). In this case just by visual observation you can tell which specimen is harder.

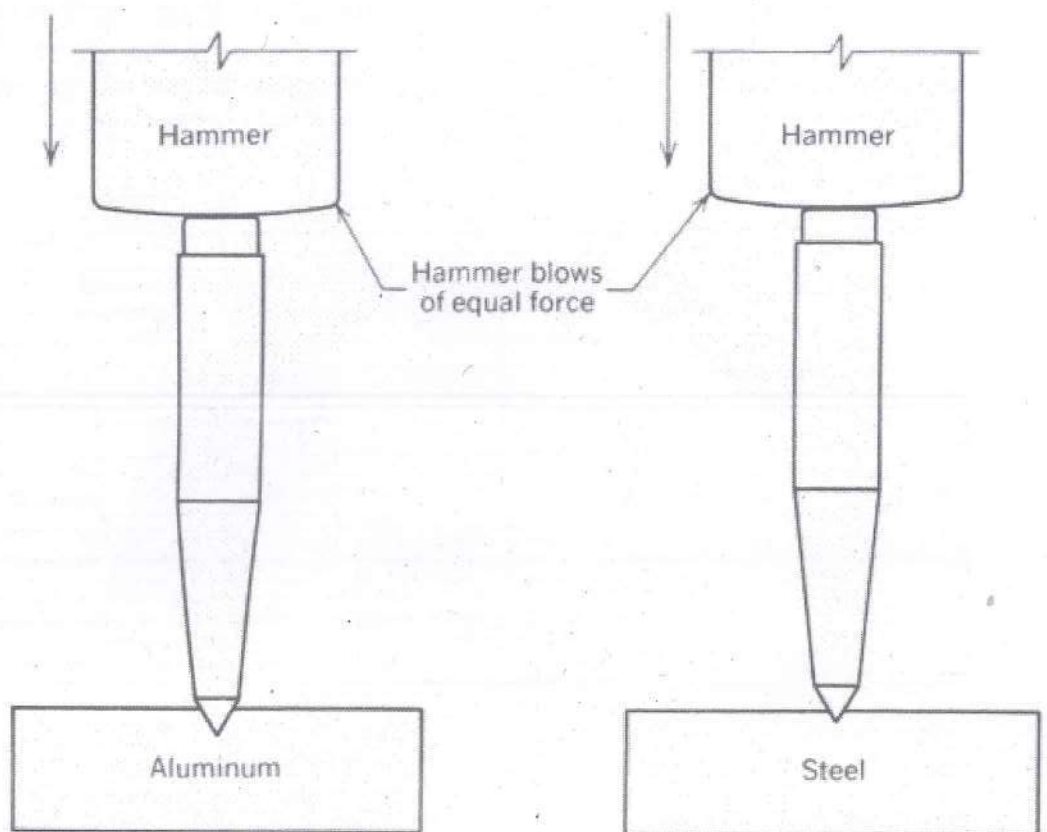


Figure 5. Indentation made by a punch in aluminum and alloy steel (Machine Tools and Machining Practice)

Wrought iron is very soft since it contains almost no carbon or any other alloying element. Generally speaking, the more carbon (up to 2 %) and other alloying elements that steel contains, the harder, stronger, and less ductile it becomes. If the steel has been annealed (softened), the hardness of a sample can help in separating low carbon steel from an alloy steel or a high carbon steel. Of course; the best way to check for hardness is with a hardness tester. The rockwell, brinell, and other types of hardness testing will be studied in one of the coming experiments. Not all machine shops have hardness testers available, in this case the following shop methods can prove useful.

Scratch Test

Geologists and "rock hounds" scratch rocks against items of known hardness for identification purposes. The same method can be used to check metals for relative hardness. Simply scratching one sample with another results in that the softer will be marked (Figures 6a and 6b). Be sure that all scales or other surface impurities have been removed before scratch testing. A variation of this method is to strike two similar edges of the two samples together. The sample receiving the deepest indentation is the softer.

The Moh scale can provide a usual means of relating one material to another in terms of relative hardness. The Moh scale hardness of some commonly used materials in the shop are in *Table 3*. The Moh scale of hardness can provide a useful means of relating one material to another in terms of relative hardness (*Table 4*).

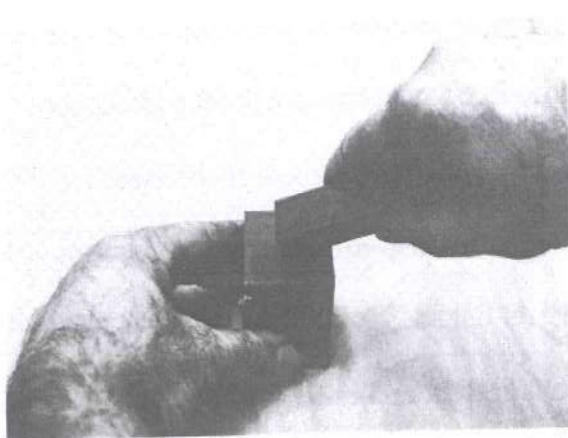


Figure 6a. a piece of mild steel is scratched across an unknown metal sample. Since the sample is not scratched, it is harder than mild steel and probably is an alloy or tool steel



Figure 6b. The sample is now scratched against the mild steel as a further test and it does scratch the mild steel

Table 3. Moh scale hardness of some commonly used materials (Callister, 2009)

<i>Material</i>	<i>Moh scale</i>
Aluminum	2 to 2.9
Brass	3 to 4
Chromium	9
Glass	5
Iron	4 to 5
Lead	1.5
Magnesium	2
Tool steel (hardened)	7.5 to 8.5
Tungsten carbide	9.5
Zinc	2.5

**Table 4. Moh scale of hardness of some commonly used materials (Callister
2009)**

<i>Number</i>	<i>Material</i>
1	Talc
2	Rock salt and gypsum
3	Calcite
4	Fluorite
5	Apatite
6	Feldspar
7	Quartz
8	Topaz
9	Corundum
10	Diamond

IV. The Experiment

The materials used in this experiment are:

- ◆ Steel, stainless steel
- ◆ Iron, galvanized iron
- ◆ Copper, brass, bronze
- ◆ Lead, tin, solder.

Used equipment

Hack saws, files, weights and weight pans, vises, pliers, magnets, vernier caliper, micrometers and chemicals.

Experimental procedure

Perform the following tests for one group at a time and compare the results for each metal in that group.

- ◆ Cutability: cut samples of 6cm length using hack saw.
- ◆ Density: weigh each sample, measure its dimensions, then calculate the density.
- ◆ Magnetism: test each sample with a magnet, is it magnetic?
- ◆ Hardness: scratch the surface of each sample with a file.
- ◆ Impact (brittleness): hold the sample in a vice with 5cm protruding from the jaws. Drop a 100g mass from a height of 1.5 m and observe.
- ◆ Fatigue: hold the sample with pliers at each end, leaving 3cm of it between the jaws. Bend the sample backward and forward counting how many bends it takes to cause a fracture.
- ◆ Strength: hold a sample in a vice, hold the other end with pliers and pull to cause failure, can you?

- ◆ Corrosion: place each sample in a beaker of dilute hydrochloric acid for 30 min. and observe.

Note: For each test rank your samples as follows: softest to hardest, easy to difficult, poor to good, low to high, as 1 to 10.

Student Notes

Questions:

Which Samples are pure metals and which are alloys?

What is the difference between pure metals and alloys?

EXPERIMENT No.2

Macroscopic examination

(Macro-etching and S-printing)

I. Purpose :

The student will select the etchant, examine and photograph the surface of a metal specimen for the purpose of revealing surface structure, defects or inhomogeneities.

IV. Background :

Review the sections in your materials science textbook covering the subject : practical metallography.

III. Introduction:

Macroscopic examination is distinguished from conventional metallographic examination by the low magnification employed and the large areas examined. Low magnification (usually not over 10x) is employed to show the entire section of a large part that may be examined rather than macroscopic areas at one or more locations.

Macroscopic examinations reveal defects and inhomogeneities over a broad area. It can be used to find the size and distribution of non metallic inclusions ; uniformity of a cast, welded, rolled or forged products, and the presence of fabricating defects such as seams, folds, flow lines or bursts, and residual ingot defects such as pipe, porosity, and other defects.

Macroetching and subsequent examinations of parts of various stages of fabrication is a widely used quality control and process improvement measure for steel and non-ferrous metals and alloys.

Selection of a specimen for macroscopic examination should be made with proper consideration of the ultimate aim of the test. For example, the cross-section of a

welded structure, a casting , or a forging products structure shows marked structural differences, where as the cross-section of a machine- cut shaft is usual quiet uniform. Heterogeneous conditions in a metal structure revealed by macroetching may be classed into one or more of the following :

- ◆ *Heterogeneous Grain Structure* : such structures are results of different solidification rates , crystal growth, gas evolvement, or thermal processing
- ◆ *Heterogeneous Chemical Composition* : these variations result from segregation due to impurities in the metal or from surface treatments such as carbonizing.
- ◆ *Heterogeneous Mechanical Properties*: arising from plastic deformation in forged or rolled parts. Such properties greatly affect structure and performance.

Thin slaps or sections of the specimen may be obtained by shearing, sawing, flame cutting, or machining which ends with irregular surface of the product. It is important that these irregularities be removed by filing or grinding to minimize obliteration of surface details as revealed by etching. The part to be examined should be kept cooled while cutting and grinding to prevent structural changes which may lead to misinterpretation of the macrostructure. Adhering oil or grease must be removed to ensure rapid and uniform etching.

The idea behind macroetching is that inhomogeneties in a metal or alloy react at inherently different rates. Consequently surface defects, inclusion segregated areas , and other types of flaws are selectively dissolved by the etching reagent and made manifest as a cavity, striation , local discoloration , or other structural characteristics. Some of these defects may be quit small and escape detection prior to macroetching during etching , however, the area occupied by the flaws will increase in size and

eventually become visible to the unaided eye. For example , the cavity occupied by a manganese sulfide inclusion in steel may be enlarged during macroetching by a factor of tow to three handred times, with a possibility that it may be incorrectly interpreted as porosity or sponginess

Etching reagents for iron or steel are generally dilute solution of one or more inorganic acids. A solution consisting of 38% hydrochloric acid 12% sulfuric acid and 50% water is recommended for general use. Other reagents are given in Table 1.

Etching rate is highly temperature-dependent. High temperature may greatly accelerate etching. The precise influence of etching temperature varies with alloy composition, heat treatment, specimen surface condition and other factors. They should be kept constant for each alloy if comparative results are wanted. In addition to etchant type and temperature etching time is also important variable in macroetching. Prologed etching may darken the etched surface and obscure or obliterate certain parts of the structure. Under-etching may not be completely developing the microstructure to reveal sufficient detail to permit accurate interpretation of the test.

When interpreting the surface characteristics revealed by macro etching, it is important to learn as much about its past history as possible, including its methods of manufacture, processing methods, thermal treatments, and service conditions and environment.

Recording of the revealed macrostructure is commonly done photographically. A number of cameras for macro photography are commercially available. The camera can be fitted with interchangeable accessory lenses to permit enlargements of up to 10x. Polaroid, roll film or regular cut film back is available, as is a 35mm back. Television camera can also be fitted above the optical tube of the microscope and

couple to a TV set (Fig.1). Illumination includes four 75-watt flood lamps set at 45 degrees to the lens-axis to reduce glare.

Table 1. Macroetchants used for macrostructural preparation of iron, steel, & cast iron (Petzow, 1978)

No.	Etchant	Condtions		Remarks
Fe M1	Ethanol (96%) Nitric acid (1.40) (Concentration variable)	90 ml 10m	1-5 min. Deep etching. zones.	Versatile, useful for Fe and Steel. Carburized, or decarbur ized Segregation. Also useful for microetching.
Fe M2	Dist. water Hydrochloric acid (1.19) (Concentration variable)	50 ml 50 ml	10-30min. up to 80°C (180°F). Remove film under running water. For corrosion-resistant steels, immerse in warm, 20% aqueous sol. of nitric acid (1.40).	Versatile etchant for alloyed and unalloyed steels. Deep etchant for surface control and segregation. Porosity hardness indentations fractures, inclusions, dendrites, flow lins, ferrite.
Fe M3	Hydrochloric acid (1.19) Sat. Aqueous sol. of Copper (II) Sulfate (Marble's reagent)	50 ml 25 ml	Secs to mins.	Austenitic and high temperature steels. Fe-CrNi cast alloys. Depth of nitriding.
Fe M4	ist. water Hydrochloric acid (1.19) Copper (II) chloride	100 ml 120-180ml 45 - 90 g	5-20 min. Prior to etching, heat (300-400°F) for 5-30 min After etching, immerse in conc. hydrochloric acid, rinse in water, and neu tralize in ammonia water.	Flow lins in low-car bon N2, steels. Thomas at 150 - 200°C steel. Compare microetchant Fe m13.
Fe M5	Dist. water Sulfuric acid (1.84) (Sulfur-printing test) (Baumann print)	100ml 5 ml	Silver bromide paper is saturated with solution, and firmly prssed against the prepared surface After 1-5 min, rinse, fix (6 g sodium thiosulfate in 100ml water), wash and dry.	Verification, arrangement, and distribution of Fe and Mn sulfide inclusions. Sulfide reacts with sulfuric acid, forming hydrogen sulfide which combines to darkish silver sulfide with the silver from the silver bromide in the photographic paper.
Fe M6	Dist. water Ethanol (96%) Hydrochloric acid (1.19) Iron (III) chloride Tin (II) chloride Add hydrochloric acid last (Oberthoffer's reagent)	500 ml 500 ml 42 ml 30g 0.5 g	Secs to mins. Microsection must be polished. After etching, rinse a 4: 1 mixture of eth anol and hydrochloric acid.	Steels and special steels. Fe-enriched areas appear dark. Blowhole segregation Primary structure. Fiber orientation.
Fe M7	Dist. water Copper (II) ammonium chloride (Heyn's reagent)	120 ml 20g	2-10min Cu precipitates wiped with water. Primary structure.	Phosphorus segregation in low-carbon steels. Fiber orientation. Welding zones, Grain contrast.
Fe M8	Sat. Aqueous sol. of sodium thiosulfate (Concentration variable) (klemm's reagent)	50 ml 1 g	Secs to mins.	Phosphorus distribution in cast steel and cast iron.

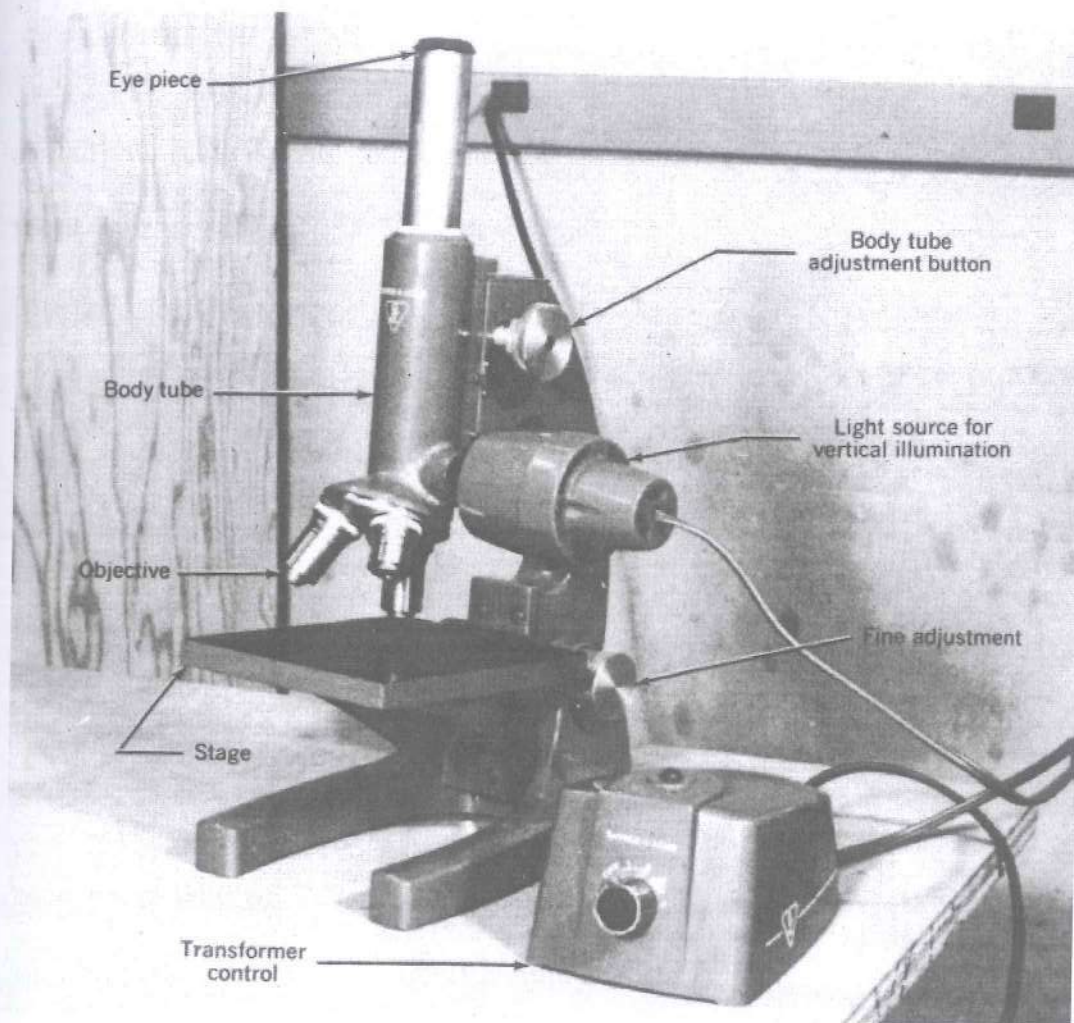


Figure 1. Metallurgical microscope

Macroscopic interpretation

Few representative types of characteristics revealed by macro etching are shown in the following figures:

- ♦ Fig.2 shows the forged structure of aluminum 2014 forged specimen. Flow lines are clearly visible.
- ♦ Fig.3 shows the macrostructure of a small cast ingot of commercially pure aluminum.

♦ Fig.4 shows the traverse section of water-quenched bar, macro-etched to reveal the depth of hardening. It is noted that the hardened case produced by quenching from an appropriate temperature etches more lightly, and thus is readily distinguished from the more severely etched core of somewhat lower hardness.

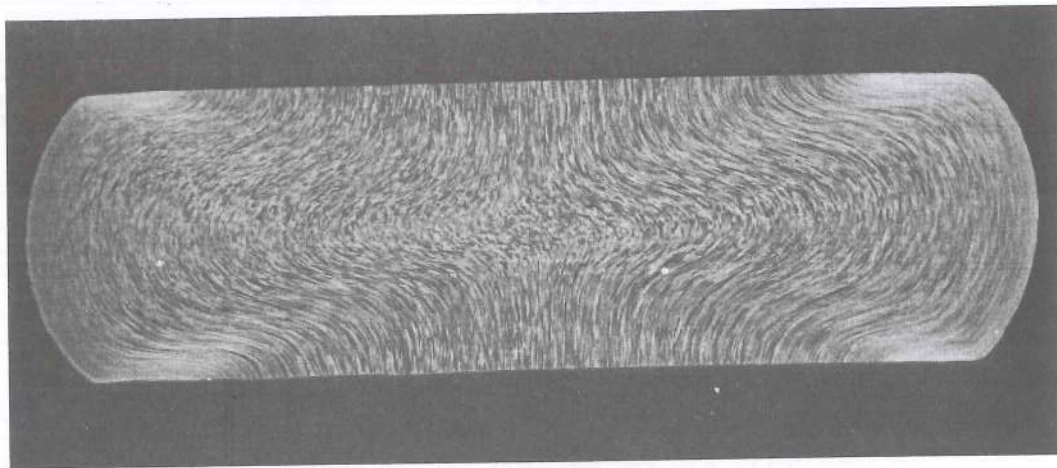


Figure 2. Forged structure of aluminum 2014 specimen. Flow lines are clearly visible. (Jalham, 1996).

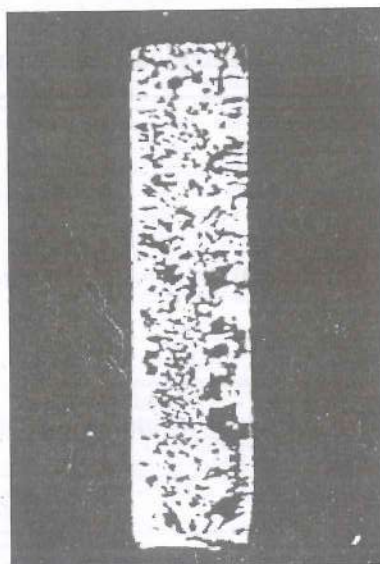


Figure 3. The macrostructure of a small cast ingot of commercially pure aluminum. (Ref. Courtesy of Messers. Metallurgical Services Laboratories Ltd., Betchworth, Surrey)

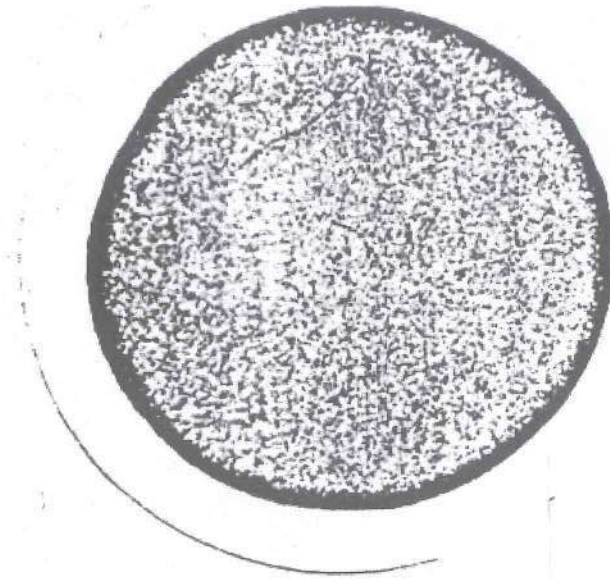


Figure 4. Cross section through a carburised bar with a case depth of 1mm.

(Higgins, 1993).

Macro etching is a convenient method of determining with some accuracy the traverse depth of a carburized case in annealed and normalized steels. The carburized case being of higher carbon content than underlying base metal will etch more rapidly and will after etching appear darker than the core. In like manner, decarburized surface may be detected by macro etching; and because these areas are very low in carbon content, and hence, etch relatively light, they may be readily distinguished.

Internal cracks, sometimes called flakes, hairline cracks or cooling cracks can also be detected as shown in Fig.5.

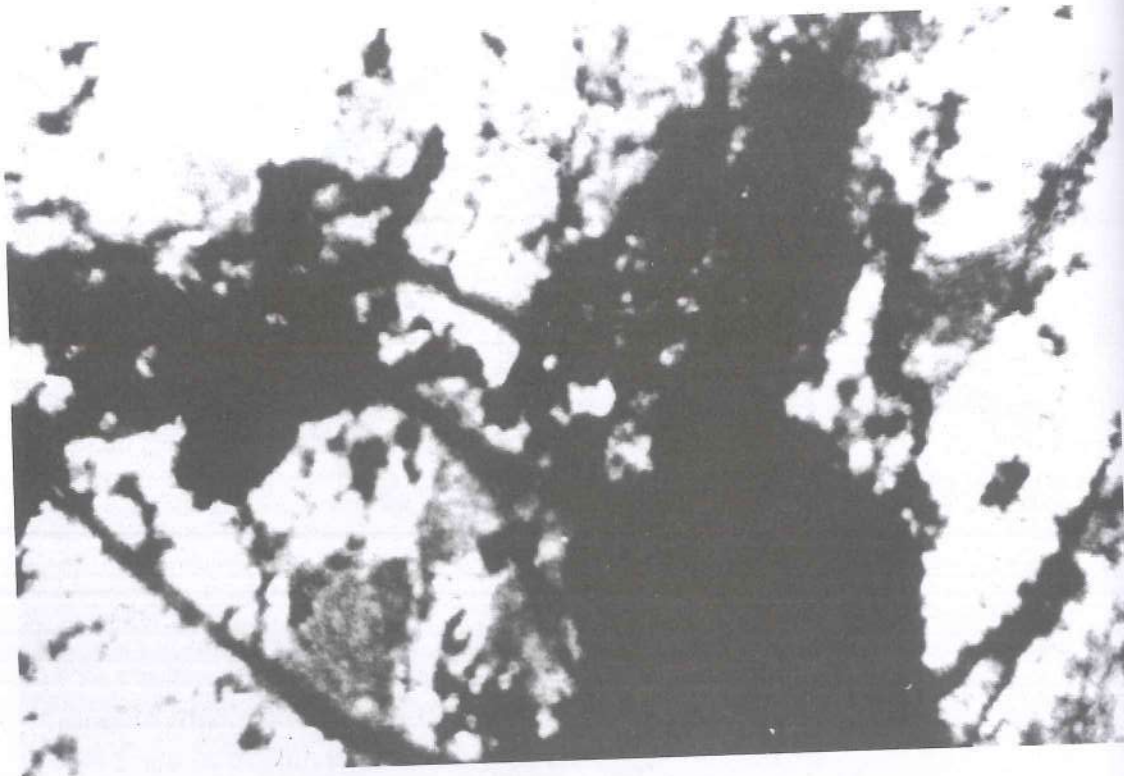


Figure 5. Internal cracks of hot forged Al-2014. (Jalham, 1996)

Sulfur printing, is used for detecting and recording the distribution of sulfur in steel. This will be discussed later in this experiment.

Caution: protect yourself and your clothing from contacting the concentrated reagents by means of a protective jace shield, rubber gloves and a protective apron.

IV. The Experiment

Material

The materials required for this experiment might include selected weldments, castings, cold formed and forged parts, and various chemical reagents as given in Table 1.

- ◆ Silver bromide photographic papers.
- ◆ 2-5% solution of H_2SO_4 .
- ◆ Photographic fixing solution.

Apparatus

The apparatus required includes saws for cutting the specimen, and an abrasive belt machine for producing a smooth surface. Etching apparatus includes a hot plate, glass thermometer, Pyrex etching bowls or breakers, stainless steel tongs, rubber gloves, face shield, fume hood, soft-bristled brush and a hot air dryer.

Equipment for recording results may include a macro camera, acrylic spray and exposure meter.

Procedure

The normal procedure for macro-examination involves the following steps:

- ◆ Obtain a specimen from the instructor.
- ◆ Cut or section the specimen if necessary. Clean all adhering dirt or grease with a suitable solvent.
- ◆ Prepare a flat surface using the abrasive belt machine or file.
- ◆ Select and prepare the proper etching solution in the beaker and if necessary, bring it to the recommended temperature on the hot plate. Use the glass thermometer for measuring temperature.

- ◆ Slowly and carefully immerse the specimen in the solution using stainless steel tongs.
- ◆ Etch the specimen for a few minutes. Remove wash carefully and examine the progress of the etch. Too short a time will not allow detail of the structure to completely develop. Too long an etching time may obliterate the structure. Continue etching the specimen until the desired results are obtained.
- ◆ Remove the specimen from the reagent; wash in running water. During rinsing, carefully scrub the specimen surface with a soft-bristle brush to remove deposits formed on the specimen during etching. Rinse the specimen in alcohol to remove last traces of water and dry on hotplate or in a warm air blast. If the specimen is to be preserved, coat it with a light coat of clear acrylic spray.
- ◆ Record on the data sheet the details of the procedure followed, including etchant, time and temperature.
- ◆ Photograph the specimen using the macro camera and attach the print to the data sheet.

Photomicrography

There are a number of excellent industrial cameras available for photomicrography, including the Polaroid MP-3 multipurpose industrial view camera, the Leitz Aristophot, the Bausch and Lomb Model L and others.

An excellent handbook on photomicrography and macro-graphy is available from Eastman Kodak; Rochester N.Y. 1962 entitled, "Photomicrography".

Photographic procedure generally involves the following steps (operating instruction for the particular camera should be read prior to using the equipment):

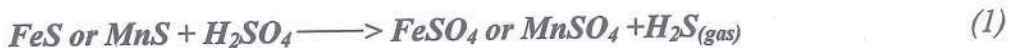
- 1- Select the proper lens combination for the magnification desired.
- 2- Position the specimen on the camera baseboard for the area to be photographed.
- 3- Sharply focus the lens on the specimen at maximum f-stop opening. Adjust lighting so the specimen is well illuminated and does not glare.
- 4- Using the light meter determine the exposure time and f-stop for the film being used, normally F-stop should be between f/22 to f/32 for maximum depth of focus. Set F-stop and cock camera.
- 5- Slide film holder into position and pull out slide.
- 6- Expose, develop and fix print ready for mounting.

If Polaroid film is used a slightly longer development time (20-30 seconds total) will improve contrast of black and white film.

Sulfur printing

Macroanalysis by sulfur printing should be done (as described below) to determine the degree of segregation. Before using another reagent, the surface must be prepared again from the beginning. Grinding the surface with fine emery paper is sufficient to remove traces of the previous etching. The section must be ground in two perpendicular directions to remove scratches.

The S-printing consists in applying ordinary silver bromide photographic paper, soaked in a 2-5% H_2SO_4 solution for 3-4 min. and then drained to the surface of the sample for 1-2 min. under moderately applied pressure. The photographic paper is removed from the surface, rinsed in clear running water and then fixed permanently by placing it in a photographic fixing solution for about 15min. After that the print is again washed in running water for approximately 30min. and subsequently dried in the usual manner. At the sulfur rich areas, the paper will darken due to formation of Ag_2S . The reactions are:



Then



Results and conclusions

Examination of the photograph or actual specimen may reveal certain characteristics which account for failure in service or which may lead to failure. Although it is not possible in this work to show all the possible types of inhomogeneities revealed by macro examination, the student may look for the three types of inhomogeneities discussed previously:

- ◆ Inhomogeneous grain structures.
- ◆ Inhomogeneous chemical compositions (S-distribution).
- ◆ Heterogeneous mechanical properties.

The experimental report should include a discussion of the equipment and experimental procedures used the defects revealed and interpretation of them. Significance of the defect should also be discussed as it relates to failure of the part to meet intended design or performance requirements.

Student Notes

Macroscopic Examination

<i>Name</i>		<i>Section</i>	
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Results:

The composition of macroetchant solution used in this experiment

Etchant composition	Time needed	Temperature

Sulfur printing photograph paper

Questions:

How did you choose the etchant to reveal the macrostructure?

What are the reasons for inhomogeneity in grain structure, chemical composition, and mechanical properties?

Show the inhomogeneity in grain structure and chemical composition on the photograph you printed in this experiment.

EXPERIMENT No.3

Metallographic Specimen Preparation For Microscopic Examination

I. Purpose :

During this experiment the student will select, mount, grind, polish , and etch metal specimens for metallographic examination.

II. Background :

Review the sections in your materials science textbook covering the subject : practical metallography.

III. Introduction :

A number of methods have been developed to reveal the micro structure of the metals and alloys. Probably the most common and useful method is microscopic examination.

Under the trained eye, such characteristics are revealed as: location, type and distribution of nonmetallic inclusions, grain size, degree of cold working, segregation, finishing temperatures, and cooling rates. In the microstructure of metals, there exists an almost complete history of mechanical and thermal treatment of the metal under examination. From information gained during microscopic examination, it is possible to predict the behavior of a metal for a given application.

Metallographic examination is of a particular usefulness when careful considerations have been given to the selection and preparation of the specimens to be examined. The various steps involved in Metallographic specimen preparation are described below.

Specimen selection

The specimen to be examined must be representative of the metal to be studied. If a structural member has failed in service and the cause for failure is to be ascertained, the specimen is to be taken from a spot where the failure originated. Both longitudinal and transverse sections are to be prepared for examination.

Longitudinal samples can reveal the length and type of inclusions. Silicate and manganese sulfide type inclusions tend to elongate readily at rolling temperatures, while the shape of alumina and globular oxide type inclusions are not or little affected. Transverse can reveal the density of such inclusions. Specimens for grain size determination should be taken parallel to the rolling or working direction so that cold working and recrystallization may be revealed.

Heat treatment specimens should include a portion of the outer skin so that decarburization or other surface conditions can be studied.

Specimen cut-off

Once the specimen to be examined has been selected, it may be cut either by saw, abrasive wheel, electrical discharge machine, or other suitable method. The abrasive cut-off machine (Fig.1) is quite versatile and may be used for cutting either soft or hardened steel depending upon the abrasive wheel used.

The abrasive wheel may be long term consumable (Fig.2) or consumable (Fig.3). It should be noted that the choice of the wheel is influenced by the hardness and ductility of the material to be cut because wheels that are too hard tend to glaze and burn the specimen while wheel that are too soft tend to wear rapidly.



Figure 1. Two types of Cut-off machines(Metallog Guide, 1992)

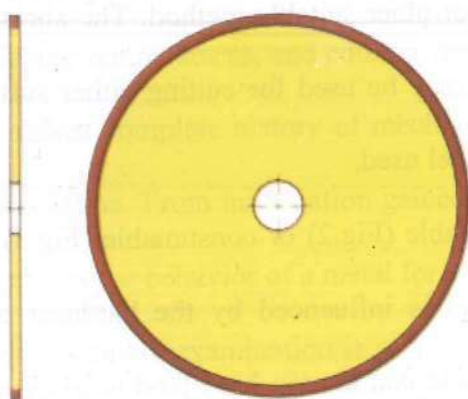


Figure 2. Long term consumable wheel. (Metallog Guide, 1992)

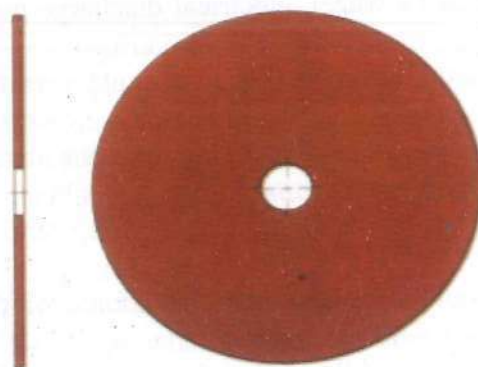


Figure3. Consumable wheel. (Metallog Guide, 1992)

It is recommended to use diamond wheels to cut ceramic and sintered carbide materials, aluminum oxide wheels to cut ferrous materials and silicon carbide wheels to cut non-ferrous alloys. Water cooling is usually employed to reduce overheating.

Cutting and all subsequent grinding and polishing operations are to be done in such a way as to avoid overheating of the specimen. Overheating may cause tempering of a hardened steel, air hardening of certain alloys, or perhaps annealing of the specimen. As a result, the structural characteristics are not representative of the original specimen and are thus valueless. It should be noted that the

Specimen mounting

A specimen which is small in size or has an irregular shape is frequently mounted in thermosetting plastic such as bakelite. Fragile specimens may be potted in commercial casting resins with glass filler to control uniformity of grinding. Clear mounting thermoplastics, such as Lucite, makes it possible to see the orientation of the specimen during grinding and polishing.

There are two Mounting techniques :

1. hot compression mounting
2. cold mounting.

For materials that have low melting points, it is recommended to use cold mounting in order to avoid microstructural changes that result from heating when using hot mounting.

In hot mounting, the specimen mounting (if required) may be easily accomplished using a press machine either (1'') or (1/4'') diameter mounts are customarily used. The press (Fig.4), which may be used with both thermosetting and thermoplastic resins, consists of a heating unit for softening the plastic and a

hydraulic ram for applying pressure during the curing or polymerization of the resin and for ejecting the cured specimen mount.



Figure 4. Automatic Mounting Press for Hot Mounting Purposes.

When thermosetting materials are used, the mold can be kept hot and curing time is short (3-4 min.). Thermoplastic materials, on the other hand, must be heated to soften the plastic, then the chamber must be cooled while maintaining pressure after which the specimen may be ejected. Cycle time in this instant may be as much as 15-20 min. Improper molding pressure or temperature or improperly shaped specimens may result in defective mounts. Thermosetting plastics such as bakelite will have a soft, gravel-like appearance if they have not been adequately heated, or if pressure is not brought up as the plastic polymerizes. Bugs may occur if the pressure is not maintained while the plastic is curing. Clear thermoplastic materials which are not heated through may have a cloudy appearance due to un-melted particles, or if not

cooled rapidly enough after melting may result in loss of transparency. Some recommended materials to be used in hot mounting are shown in Table 1.

Table 1. Recommended materials for hot mounting. (Metalog Guide 1992)

Resin	Application	Specific property	Material
ConduFast	Electropolishing	Electrically conductive Very low shrinkage	Acrylic resin with iron powder filler, Thermoplastic, Gray
SpeciFast	Glass clear mounts Porous samples Surface electrical insulator for ConduFast	Transparent Medium shrinkage	Acrylic Thermoplastic
PolyFast	Edge retention	Very low shrinkage Medium removal rate	Phenolic resin with carbon filler Thermosetting, Black
	Examination in scanning electron microscope	Electrically conductive Low emission in the vacuum chamber	
IsoFast	Edge retention Planeness	Low shrinkage Low removal rate	Diallylphtalate with glass fibre filler Thermosetting, Green
DuroFast	Edge retention Planeness	Very low shrinkage Good adhesion Very low removal rate	Epoxy with mineral filler Thermosetting, Black
MultiFast	Routine examination Backup resin	Low shrinkage Medium removal rate	Phenolic resin with wood flour filler, Thermosetting, Black
MultiFast Green	Routine examination Colour marking	Low shrinkage Medium removal rate	Phenolic resin with wood flour filler, Thermosetting, Green
MultiFast Brown	Routine examination Colour marking	Low shrinkage Medium removal rate	Phenolic resin with wood flour filler, Thermosetting, Brown
Pre-Mount	Serial mounting of uncomplicated shapes	Easy to handle Low shrinkage Medium removal rate	Phenolic resin with wood flour filler. In tablet form Thermosetting, Black

In cold mounting, the sample is placed in a mould then a mixture of two or three components is poured over the sample. The components of the mixture are some type of mounting resin and a suitable hardening material. Epoxy, acrylic, and polyester types of resin are used in cold mounting. Some recommended materials to be used in cold mounting are shown in Table 1.

Figure 5 shows the specimen after mounting regardless using hot or cold mounting.

Table 2. Recommended materials for cold mounting. (*Metalog Guide 1992*).

Name	Application	Features	Curing time	Mounting cups
SpeciFix-20	Epoxy	Vacuum impregnation Porous samples Mineralogical samples	8 h.	Multiform
SpeciFix-40	Epoxy	Vacuum impregnation Porous samples Mineralogical samples	3.5 h. at 50 °C	Multiform
Epofix	Epoxy	Vacuum impregnation Porous samples Mineralogical samples	8 h.	Multiform
Caldofix	Epoxy	Vacuum impregnation Porous samples Mineralogical samples	1.5 – 2 h. at 70-80 °C	Multiform
MetaFix-20	Acrylic	Serial mounting Irregularly shaped samples	15 min.	Multiform Flexiform Seriform
Citofix	Acrylic	Serial mounting Irregularly shaped samples	7 – 10 min.	Multiform Flexiform Seriform
Durofix-2	Acrylic Mineral filler	Serial mounting Edge retention Irregularly shaped samples	15 min.	Multiform Flexiform Seriform
Acryfix	Acrylic	For routine work	9 – 10 min.	Multiform Flexiform Seriform
Triofix-2	Polyester / Acrylic / Mineral filler	Edge retention Planeness	15 – 18 min.	Multiform Flexiform
Serifix	Polyester	Serial mounting	45 min.	Multiform Flexiform Seriform

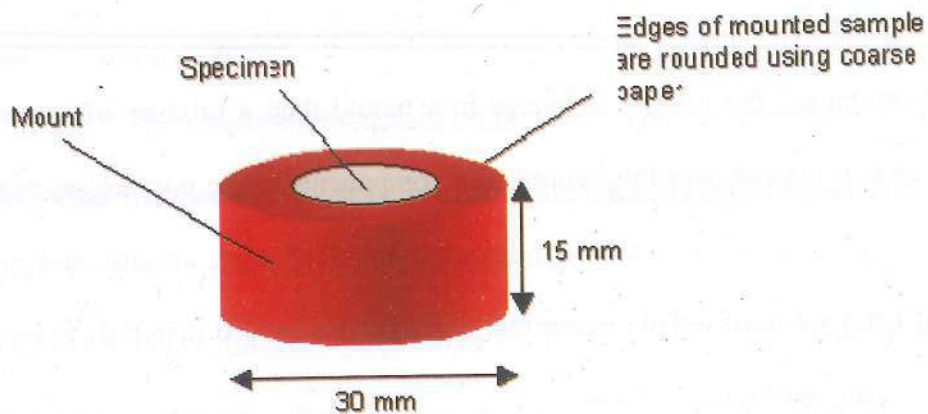


Figure 5. Atypical specimen after mounting regardless using hot or cold mounting.

Mechanical preparation

The mechanical preparation parameters for revealing the microstructure are:

- ♦ The grinding disc or polishing cloth used for preparation.
- ♦ The abrasive used for grinding and polishing.
- ♦ The grain size of the abrasive used.
- ♦ The liquid used for cooling and lubricating.
- ♦ The speed with which the grinding or polishing disc is rotating.
- ♦ The total force with which the sample holder is pressed against the grinding and polishing disc.
- ♦ The preparation time

Mechanical preparation is divided into two operations: grinding and polishing.

Grinding

During all stages of plane or coarse and fine grinding the specimen is rotated so that new finer scratches are formed at approximately 90° to those from last rotation. This makes it easy to remove the deformation which the coarse scratches have been produced by a series of new finer ones. The direction of rotation of the sample and the direction of grinding in one step and the next one are shown in Figures 5 and 6 respectively. Fine grinding is done with finer abrasive size than plane grinding. It reduces the depth of the deformed metal layer formed by coarse or plane grinding. The recommended grain size of the abrasive which is used for grinding can be determined from the curves shown in Figure 7. Atypical grinding machine is shown in Figure 8.

For very quick acceptable results, the following parameters can be used as guidelines depending on the hardness of the material. For example: the parameters shown in Table 3 are for the materials of Vickers hardness (HV) less than or equal 160. While

Tables 4 and 5 are for materials of harnesses' between 160 and 800, and higher than 800 respectively.

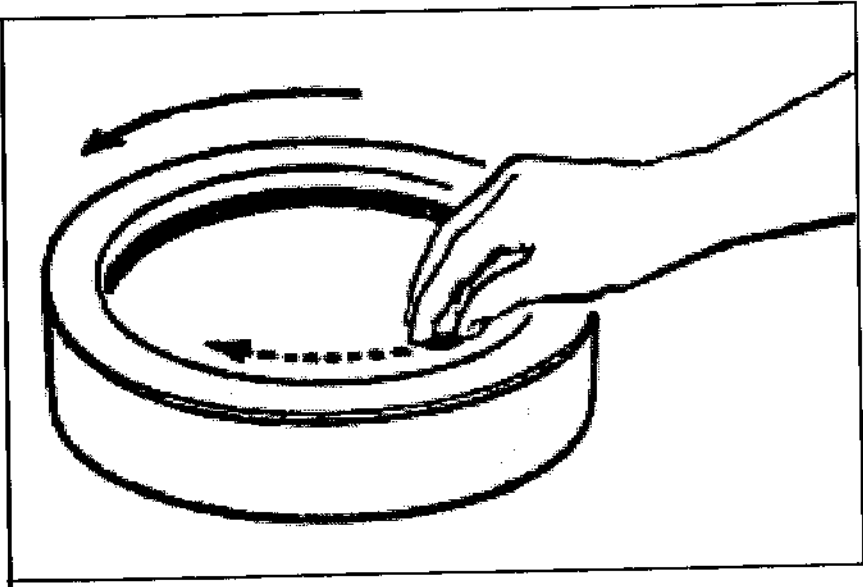


Figure 5. Direction of rotation during grinding and polishing

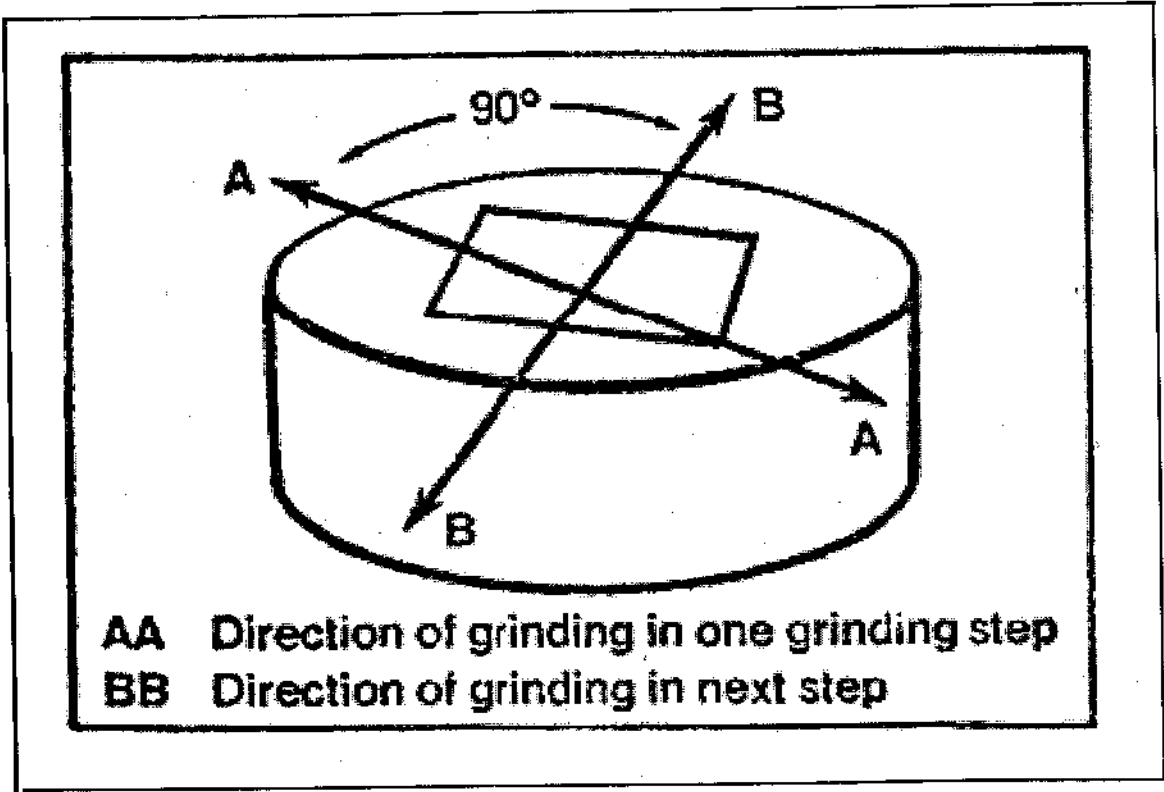


Figure 6. Grinding directions of successive steps

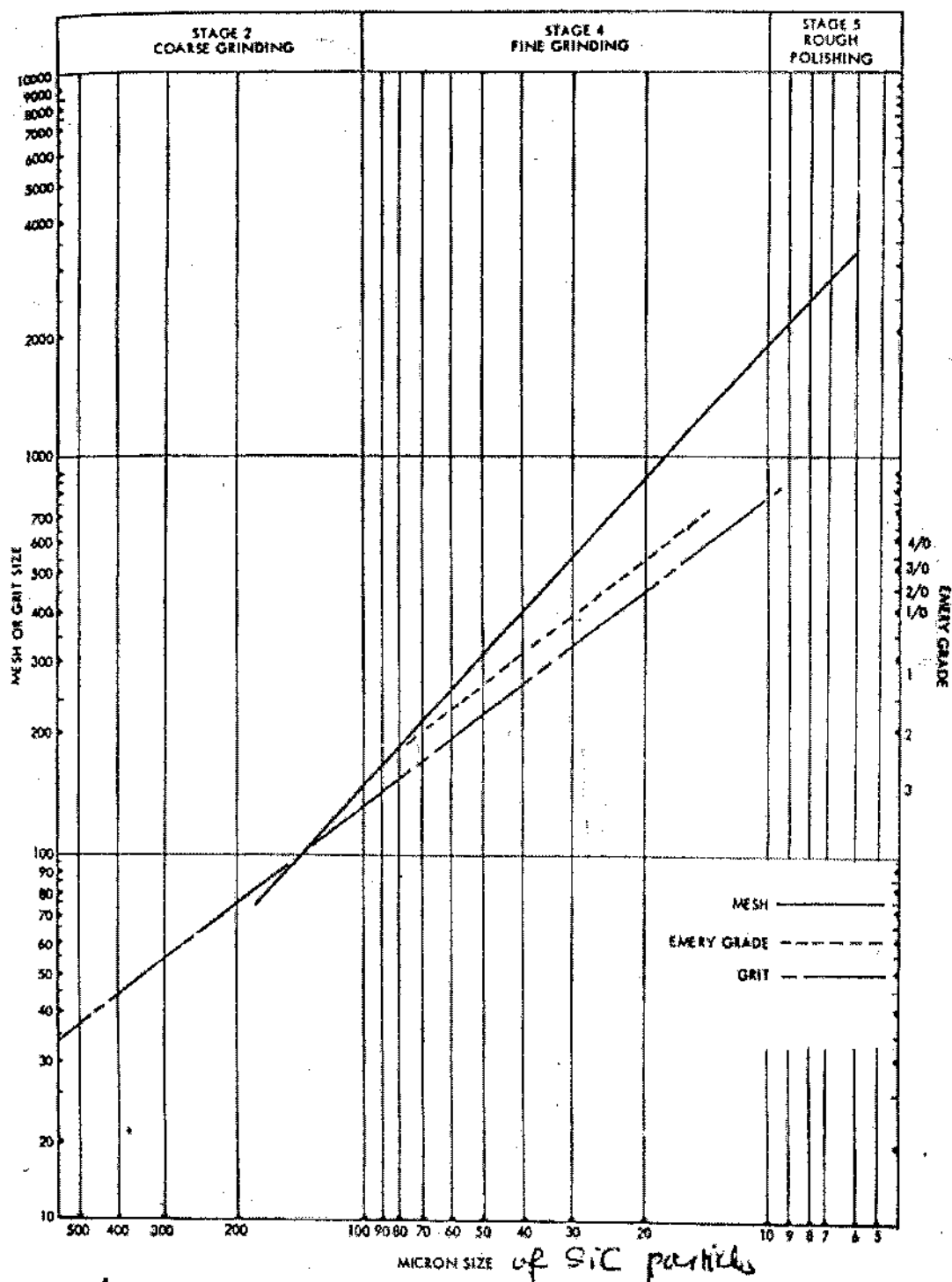


Figure 7. Relationship between grit size, mesh size, and micron size for abrasive particles. For conversion to micron size locate grit size on ordinate, transpose this point to corrected graph line and locate this position on abscissa. (Buehler Ltd)



Figure 8. A typical grinding machine

Table 3. Recommended grinding parameters for materials of HV hardness less than or equal 60. PG is plane grinding and FG is fine grinding. (Metallog Guide, 1992)

 Step	 PG	 FG
 Surface	MD-Primo 220	MD-Largo
 Abrasive	SiC	DP-Suspension
 Grit/ Grain size		9 μm
 Lubricant	Water	Green / Blue
 [rpm]	300	150
 Force [N]	120	180
 Time [min]	Until plane	5

Table 4. Recommended grinding parameters for materials of HV hardness more than 60 and less than 800. PG is plane grinding and FG is fine grinding.

(Metallog Guide, 1992)

 Step	 PG	 FG
 Surface	MD-Piano 120	MD-Allegro
 Abrasive	Diamond	DP-Suspension
 Grit/ Grain size		9 μm
 Lubricant	Water	Green / Blue
 [rpm]	300	150
 Force [N]	210	210
 Time [min]	Until plane	4

Table 5. Recommended grinding parameters for materials of HV hardness more than or equal 800. PG is plane grinding and FG is fine grinding. (Metallog

Guide, 1992)

 Step	 PG	 FG
 Surface	MD-Piano 120	MD-Allegro
 Abrasive	Diamond	DP-Suspension
 Grit/ Grain size		9 μm
 Lubricant	Water	Green / Blue
 [rpm]	300	150
 Force [N]	180	210
 Time [min]	Until plane	4

Polishing

Polishing is normally done in two steps: rough and fine polishing. One very effective polishing procedure is to use 6 micron diamond paste on a nylon or alcohol. An oil carrier is employed to distribute the diamond paste. Low speed and heavy hand pressure is employed. This results in cool, rapid, cutting with the sharp diamond grits and minimum of surface deformation. The specimen is moved in a circular motion around the wheel in a direction opposite wheel rotation.

Final polishing of the steel may be readily accomplished with 0.1 micron gamma alumina in distilled water. A micro-cloth wheel covering with low speed and fairly light hand pressure is recommended. Figure 9 shows the machine used for polishing and grinding operations. Between successive steps in grinding and polishing, the specimen should be thoroughly washed and dried using the specimen dryer shown in Figure 10 so that the coarse abrasive particles are not carried to subsequent operations. Deep scratches will result if this precaution is not taken.



Figure 9. Polishing machines.

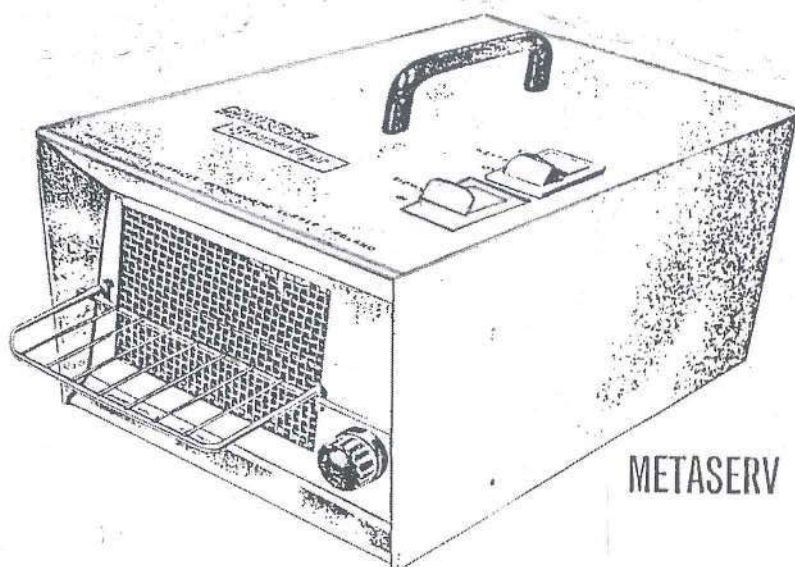


Figure 10. Specimen Dryer

For very quick acceptable results, parameters shown in Tables 6, 7, and 8 can be used as guidelines depending on the hardness of the material.

Table 6. Recommended polishing parameters for materials of HV hardness less than or equal 60. DP is diamond polishing. (Metallog Guide, 1992)

 Step	 DP
 Surface	MD-Nap
 Abrasive	DP-Suspension
 Grain size	1 μm
 Lubricant	Red
 [rpm]	150
 Force [N]	150
 Time [min]	3

Table 7. Recommended polishing parameters for materials of HV hardness more than 60 and less than 800.. DP is diamond polishing. (Metallog Guide, 1992)

 Step	 DP
 Surface	MD-Plus
 Abrasive	DP-Suspension
 Grain size	3 μm
 Lubricant	Green / Blue
 [rpm]	150
 Force [N]	180
 Time [min]	4

Table 8. Recommended polishing parameters for materials of HV hardness more than 800.. DP is diamond polishing. (Metallog Guide, 1992)

 Step	 DP
 Surface	MD-Pan
 Abrasive	DP-Suspension
 Grain size	3 μm
 Lubricant	Green / Blue
 [rpm]	150
 Force [N]	180
 Time [min]	6

Etching

During the preparation of Metallographic specimens by grinding and polishing, a layer of disturbed metal is formed on the polished surface. Electron diffraction studies reveal a disorganized and sometimes smeared metal surface extending inwardly several inter-atomic distances. Under this top layer of disorganized surface atoms is a layer of cold-worked or distributed metal of finite thickness, estimated to be 10-50 times in depth of scratch producing the deformation. It is this layer of distributed whose presence profoundly influences the appearance of the observed structure and which must be removed if the true structure to be revealed.

Chemical etching and electrolytic polishing are commonly used for removing this disturbed and deformed metal. Chemical etching effectively removes the layer by subjecting the polished surface to carefully controlled chemical attack by an appropriate reagent. When Metallographic specimen is etched, the disturbed metal may not be completely removed by a single etching treatment. It is recommended in practice to alternately and lightly polish and etch it, several times if necessary, until examination under the microscope reveals no changes in the appearance of the microstructure.

Usually three cycles of alternate etching and polishing are sufficient for all but soft metals and alloys. Electrolytic polishing is particularly valuable for this type of specimen. Etching may be done by immersing the specimen face up in a shallow beaker of the reagent or by swabbing with cotton. The etched specimen should be immediately washed in warm water.

Some recommended etching solutions to reveal the microstructure are shown in Table 9.

Table 9. Recommended microetching solution to reveal the microstructure.

(Petzow, 1978).

No.	Etchant	Conditions		Remarks
Fe m1	Ethanol or methanol (95%) Nitric acid (1.40) (Nital) See note at Fe m3.	100 ml 1-10ml	Secs to mins. Caution: not to exceed 10% solution. Explosive!	Most common etchant for pure iron, low-carbon steels, alloy steels and gray cast iron. Segregations can cause uneven attack.
Fe m2	Ethanol or methanol (95%) Hydrochloric acid (1.19) (Concentration variable)	100 ml 20ml	5-30 min. Use fresh only! Possible addition of Hydrogen peroxide (30%)	Differentiation of Cubic and tetragonal martensite. Ni-con- taining Cr steels. High-temperature steels.
Fe m3	Ethanol (96%) Picric acid (Concentration variable) (picral) Nital and Picral (Fe m1 and Fe m3) may be mixed 1:1	100 ml 2-4	Secs to mins. !!! See Appensix A.	Generally used for iron and heat treated steels. Pearlite, martensite, and bainite. Lower contrast than Fe m1. Unifrom etching. even with segrega- tions. Fe, C stained light yellow.
Fe m5	Ethanol (96%) Hydrochloric acid (1.19) Picric acid Possibly a few drops hydrogen peroxide (3%) or wetting agent	100 ml 1-5 ml 1-4g	Sects to mins. To bring out grain boundaries, heat for 10 min at 300-500°C (570-930°F) before etching !!! See Appendix A.	Increases attack of Fe m2 in alloyed steels. Grain-boundary precipitates in Cr-Ni steels steels becom visible
Fe m6	Ethanol or methanol (95%) Hydrochloric acid (1.19) Nitric acid (1.40)	Up to several mins. 85 ml 1-10 ml 1-5 ml	!!! See Appendix A.	Grain boundaries in heat treated tool steels. Alloyed Cr steels.
Fe m7	Ethanol (96%) Nitric acid (1.40) Hydrochloric acid (1.19) Picric acid Possible addition of wetting agent.	80 ml 10 ml 10 ml 1g	Secs to mins. !!! See Appendix A.	Grains boundaries in martensitic micro Structures.
Fe m8	Dist. water Sodium hydroxide Picric acid	75. ml 25g 2g	3-15 min. 50°C (120°F). !! See Appendix A.	Cementite (Fe1 C) with up to 10% Cr is stained dar. For more than 10% Cr. no standing. (Fe, Cr), Cr. (Fe, Cr)w, C2, Wc and VC are not stained.
Fe m10	Dist. water Hydrochloric acid (1.19) Nitric acid (1.40) Vogel's special reagent (V2A etchant)	100 ml 100 ml 10 ml 0.3 ml	Secs to mins, room temperature to 50°C (120°F).	Mn-alloyed Cr-Ni steels. Sigma phase and ferrite. Fine microstructures in alloyed steels.
Fe m11	Glycerol Nitric acid (1.40) Hydrochloric acid (1:19) (Concentration variable) (Vilella's reagent)	45 ml 15 ml 30 ml	Secs to mins. !!! See Appendix A.	Stainless steels with high Cr content. Cr-Ni cast steels.
Fe m12	Glycerol Nitric acid (1.40) Hydrofluoric acid (405) (Concentration variable)	20-40 ml 10 ml 20 ml	Secs to mins. !!! See Appenis A.	Steels with high Si content.

General rules:

- 1- cleaning is vital. Wash hands and sample between each grinding and polishing state.
- 2- Keep sample cool, particularly during cut-off. Heat may change the sample structure.
- 3- Rotate the sample 90° between grinding stages so the removal of previous scratches may be readily observed.
- 4- Change abrasive paper as needed : they load up rapidly when used dry.
- 5- During final polishing, a cloth that is too wet may cause pits in the specimen: if too dry, buffing and or smearing may result.

IV. The experiment

Materials

- ◆ Select ferrous specimen suitable for mounting .these may include specimens with ferrite, pearlite, austenite, martensite, bainite, spherodized or other microstructures.
- ◆ Abrasive cut-off wheels, (1/16``) thick for hard and soft steel specimens.
- ◆ Bakelite mounting powder (1 table spoon/specimen).
- ◆ Abrasive belt,or paper 120 or 180 grit silicon carbide.
- ◆ Abrasive paper 240,320,400 and 600 grit silicon carbide.
- ◆ Diamond paste,6-micron or other suitable polishing wheel covering.
- ◆ Natal etching 2% (2ml conc. Nitric acid in 98ml methyl or ethyl alcohol).

Apparatus

Several pieces of apparatus are required for metallographic specimen preparation a wide variety of apparatus is commercially available and may include the following:

- ◆ Abrasive cut-off machine, water cooled.
- ◆ Abrasive belt machine for deburring specimens and for rough grinding.
- ◆ Specimen mounting press with heater for mounting samples in bakelite.
- ◆ Vibratory marking tool for engraving identification on mounted specimen.
- ◆ Metaserv abrasive surfacer for rough and fine grinding 240,320,400 and 600 grit.
- ◆ Rotary diamond polishing unit with suitable covering.
- ◆ Metasurv final polishing unit with microloth covering.
- ◆ Ultrasonic cleaning unit.

- ◆ Hot air electric dryer for drying specimen after polishing and etching.
- ◆ Petri dishes, beakers and various glassware for etching reagent.
- ◆ Dessicant storage cabinet for protecting etched specimens.

Metallographic Specimen Preparation For Microscopic Examination

<i>Name</i>		<i>Section</i>	
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Results:

The composition of microetchant solution used in this experiment

Etchant composition	Time needed	Temperature

Photo micrograph of the used material

Questions:

How did you choose the etchant to reveal the microstructure?

Show the grain boundaries on the micro photograph you printed in this experiment.

Why did you get clear boundaries?

EXPERIMENT No.4

Plastic deformation, Recrystallization, and Grain size calculation

I. Purpose

The purpose of this experiment is to see the change in microstructure and mechanical properties of a metal after plastic deformation by measuring the hardness and calculating the grain size of a metal before and after plastic deformation.

II. Background

Review the section in your materials science text book covering strain hardening and annealing and in your note book the grain size calculation using intercept method.

III. Introduction

Plastic deformation in crystalline materials occurs primarily by dislocation motion in a shear process called slip or glide. This shear is restricted in most crystals to a single set of equivalent slip directions on only a few slip planes which contains these directions. If a shear stress is applied to a crystal in a direction other than a direction of easy shear, slip still occurs in a preferred direction even though the component of shear stress resolved onto that direction may be much lower than the applied stress.

When dislocations move during plastic deformation, they multiply and interact in ways which increase the stress required for further plastic deformation. This process is called strain or work hardening. Thus, prior plastic deformation causes the strength or hardness of a metal to increase.

Most of the work of plastic deformation is converted to heat which raises the temperature of the metal as it's being deformed. Some of the work, however, is stored in the metal as lattice defects. These defects distort the lattice and therefore have an energy associated with them. This internal energy in a deformed metal causes it to be thermally unstable. Upon heating such a distorted crystal, which increases the mobility of the lattice defects, a number of processes tending to return the crystal to its original position of perfection take place. One of these processes is recrystallization. A boundary between a distorted grain and an undistorted grain will always tend to migrate in such a direction as to extend the undistorted grain at the expense of the distorted one. At sufficiently high temperatures, the rate of migration can be rapid. Recrystallization is the formation and growth of new, more perfect grains in the distorted matrix, thus removing the effects of cold work. The first small recrystallized grains that appear are observed to form in regions of severe complex distortion, for example, near grain boundaries. It is also observed that the greater the degree of cold work, the lower is the temperature at which new grains begin to appear. This tendency can be explained in terms of the driving force for recrystallization. With greater amounts of cold work, more crystalline defects are created. The more defects there are, the greater the internal energy and, thus, the driving force for recrystallization. For this reason as well, the number of the recrystallization nuclei tends to increase, and thus the final grain size decrease, as the amount of cold work increase.

At temperatures higher than that which just gives complete recrystallization in the given time, grain growth which is a slow coarsening of the average grain size by the growth of some grains at the expense of their neighbors, occurs. Both recrystallization and grain growth tend to decrease the hardness and strength of a metal. This means

that the change in the grain size or shape influences the mechanical properties of materials. To judge that there is a change in the grain size, the grain size should be measured. There are two main methods of measuring the grain size:

- 1- The ASTM comparative method based on standard charts defining areas of grains intersecting a planar section through the microstructure. This method is at best only semi-quantitative and very subjective.
- 2- The mean linear intercept (m.l.i) method which define the average chord length intersected by the grains on a random straight line in the planar polished and etched surface.

The ASTM comparative chart method.

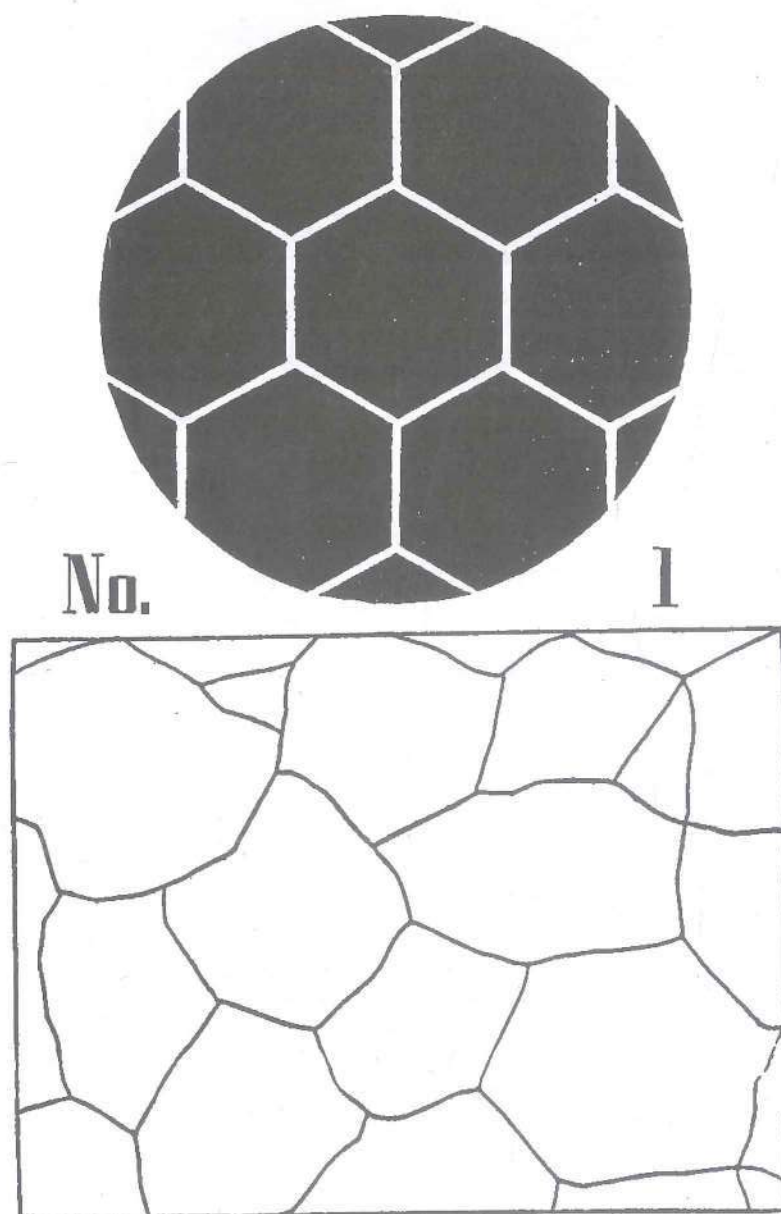
In this method, the standard chart against which the microstructure is compared comprises either photomicrographs (mesh of irregular shapes) or idealized hexagonal (mesh of regular shapes) networks based on the number of grains intersecting unit area of the plane of polish. The comparison is made by holding the standard grain size chart alongside the image of the material grain projected on ground glass screen attached to a photomicrograph. If such projection equipment is not available, the comparison can be made using a transparent ASTM network which is overlaid on the photomicrograph of the required grain structure at the proper magnification.

In this method, the grain index number is an arbitrary experimental number with reference to the mean number of grains per square inch at a magnification of 100x. Thus, if the ASTM grain size number is N , this is related to the number of grains n per square inch area and magnification 100x by:

$$n=2^{(N-1)} \dots\dots\dots(1)$$

or $N= 1 + (\log n)/(\log 2) \dots\dots\dots(2)$

The standard charts for grain size numbers from 1 to 8 are shown in Figures from 1 to 8 below.



**Figure 1. Upper, idealized regular shape hexagonal network for mean grain size no.1.
Lower, ASTM standard grain size no.1 of irregular shape mesh. Up to 1.5 gr/in² at
100x**

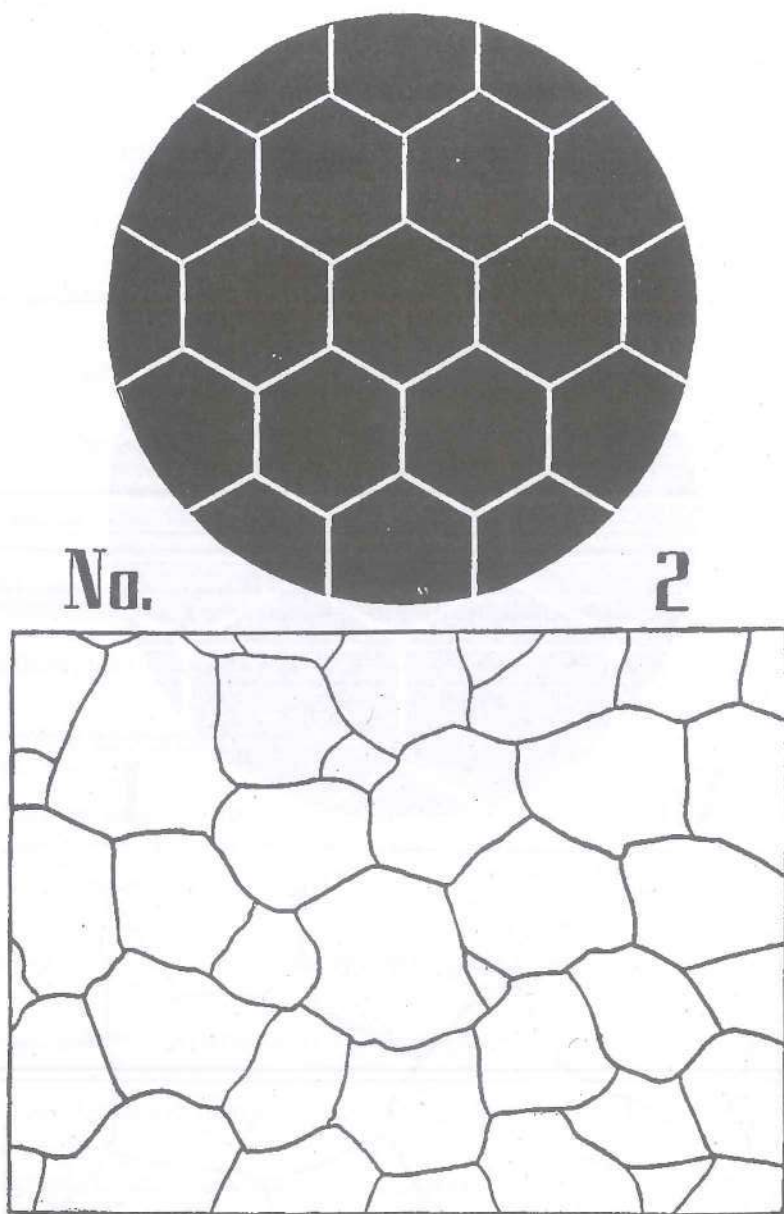


Figure 2. Upper, idealized regular shape hexagonal network for mean grain size no.2. Lower, ASTM standard grain size no.2 of irregular shape mesh. 1.5-3 gr/in² at 100x

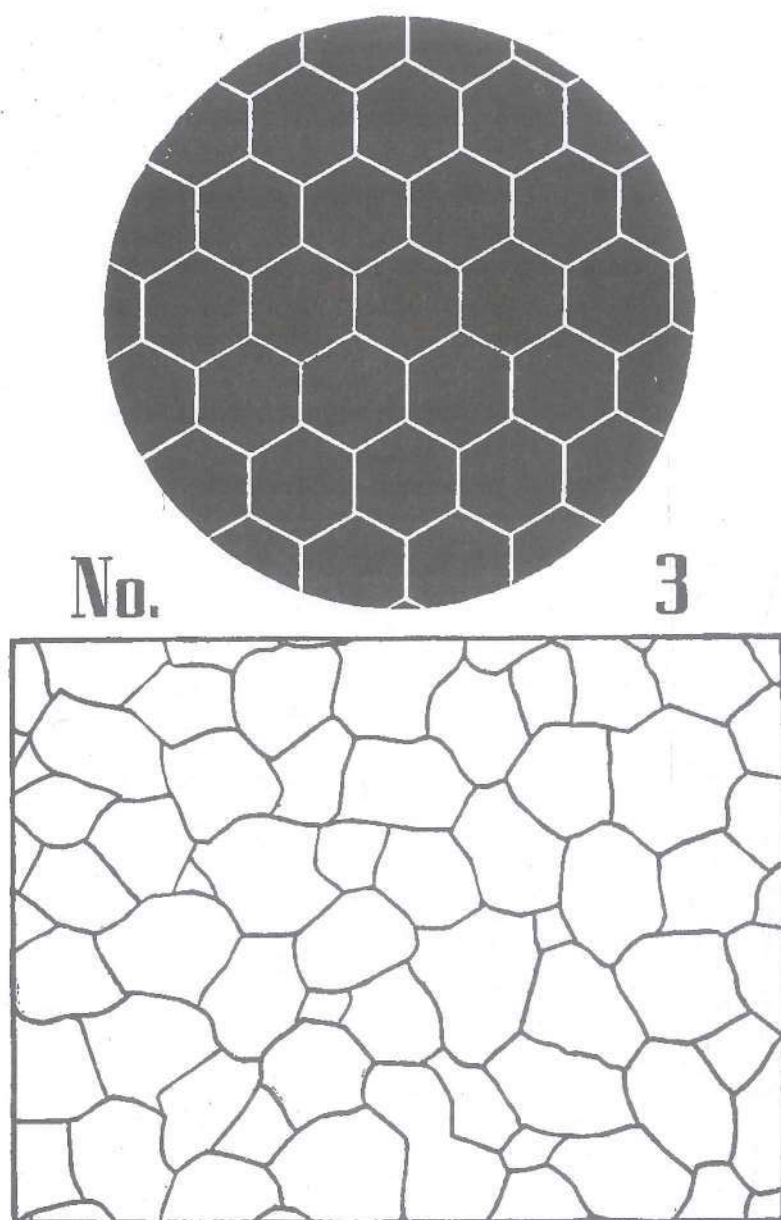


Figure 3. Upper, idealized regular shape hexagonal network for mean grain size no.3. Lower, ASTM standard grain size no.3 of irregular shape mesh. 3-6 gr/in² at 100x

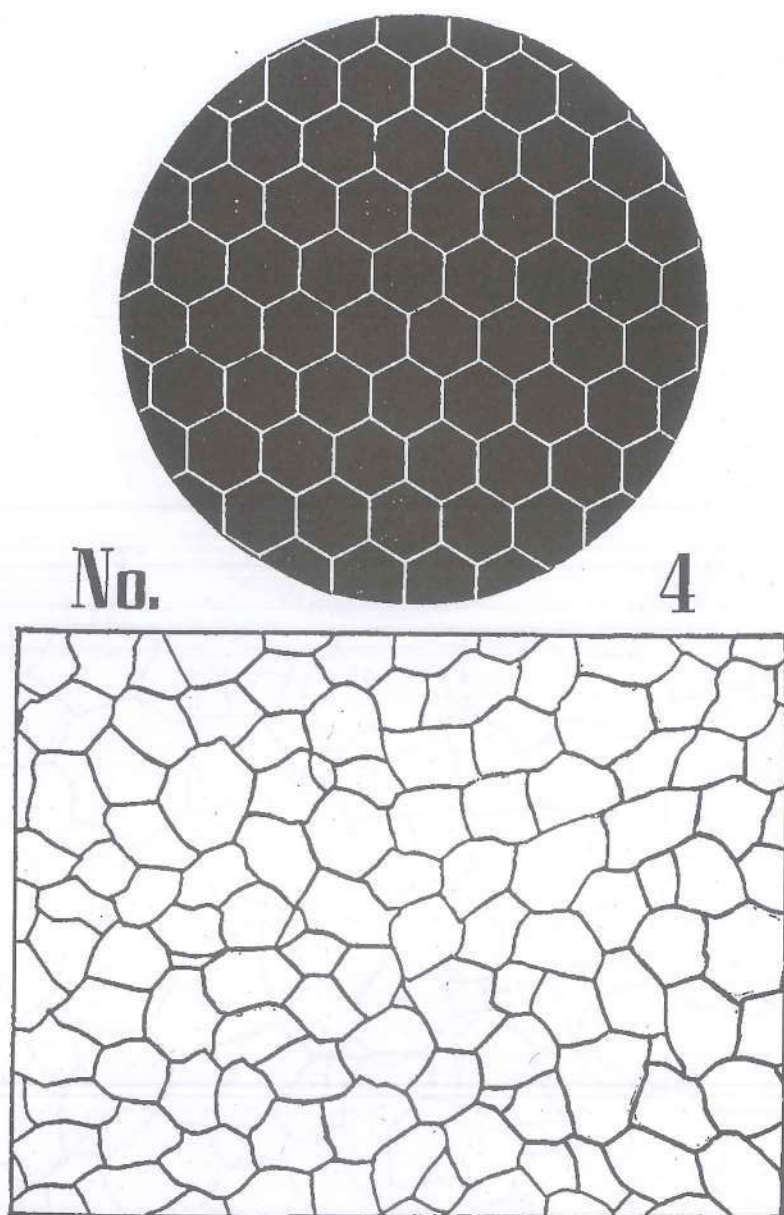


Figure 4. Upper, idealized regular shape hexagonal network for mean grain size no.4. Lower, ASTM standard grain size no.4 of irregular shape mesh. 6-12 gr/in² at 100x.

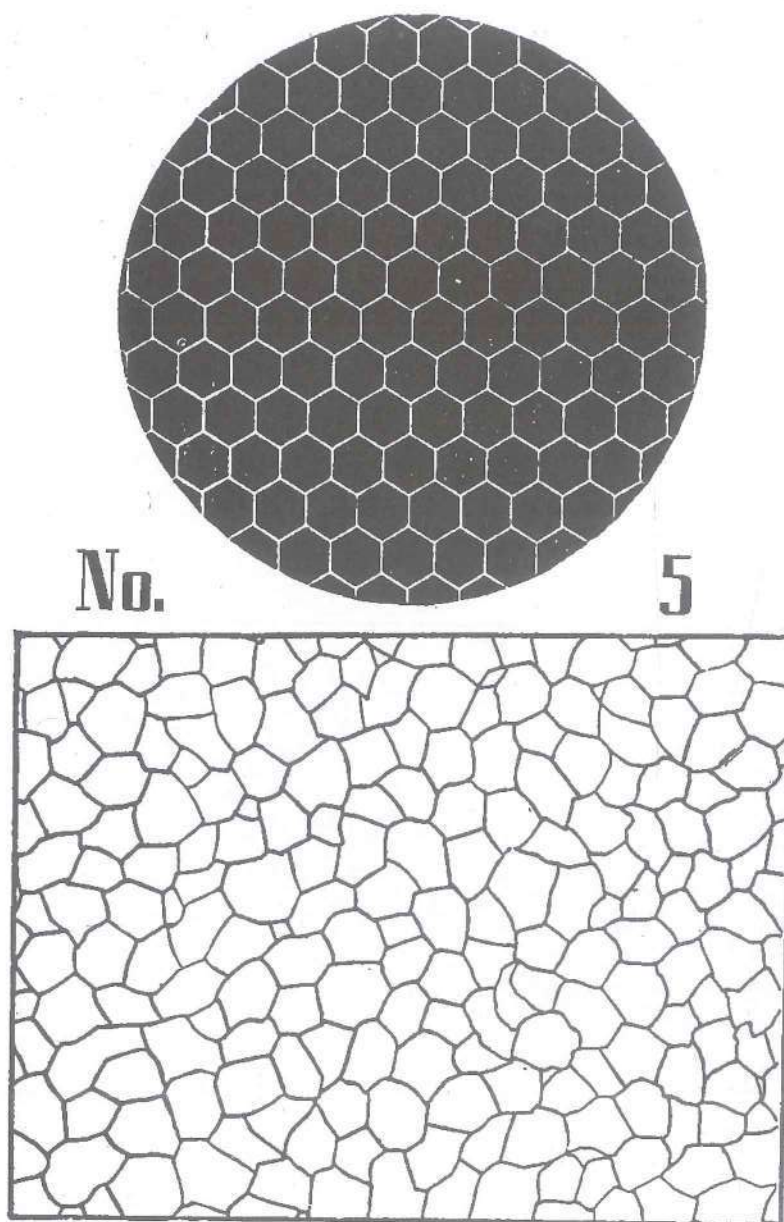


Figure 5. Upper, idealized regular shape hexagonal network for mean gran size no.5. Lower, ASTM standard grain size no.5 of irregular shape mesh. 12-24 gr/in² at 100x.

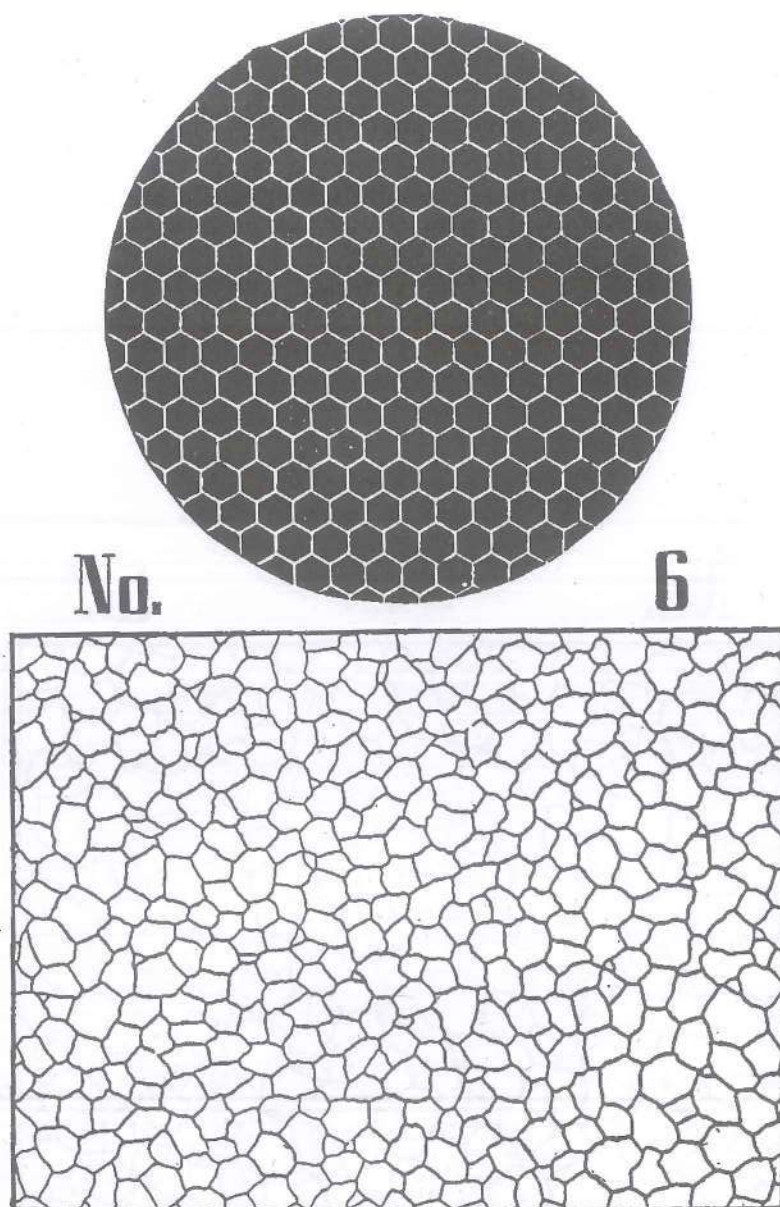


Figure 6. Upper, idealized regular shape hexagonal network for mean gran size no.6. Lower, ASTM standard grain size no.6 of irregular shape mesh. 24-48 gr/in² at 100x.

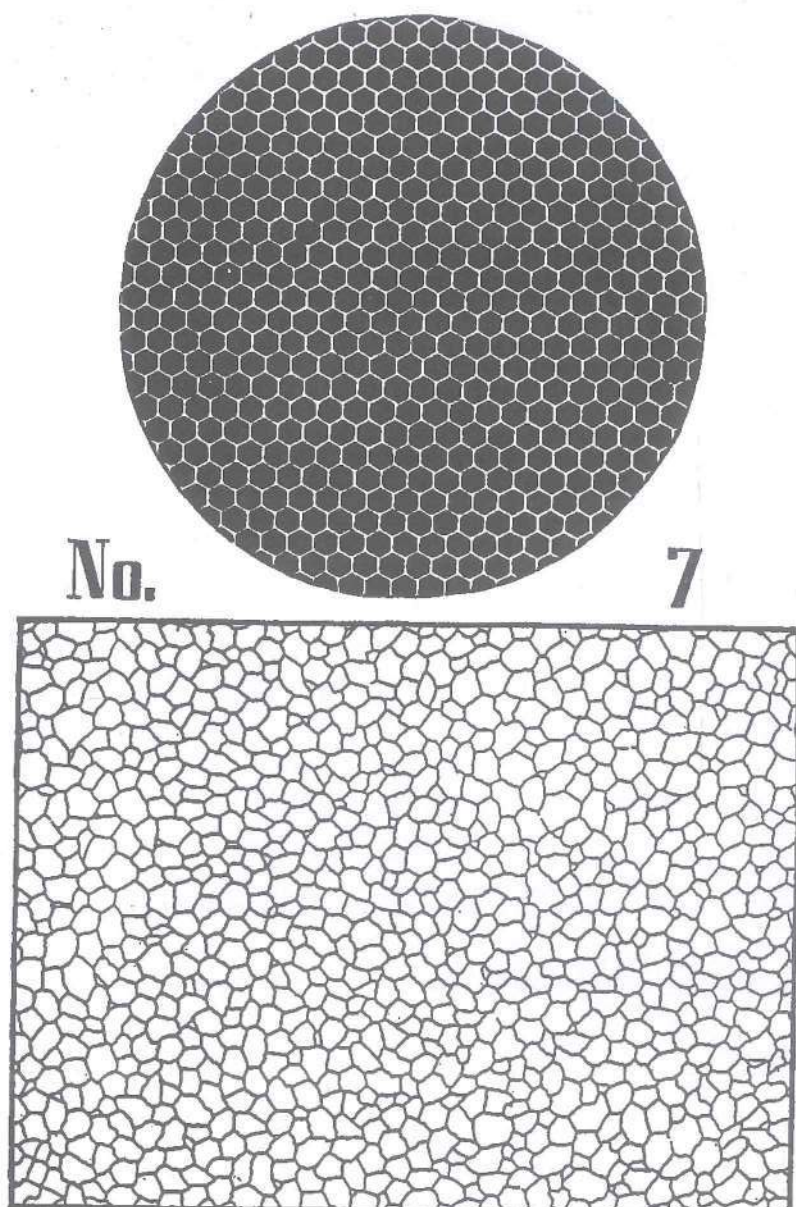


Figure 7. Upper, idealized regular shape hexagonal network for mean gran size no.7. Lower, ASTM standard grain size no.7 of irregular shape mesh. 48-96 gr/in² at 100x.

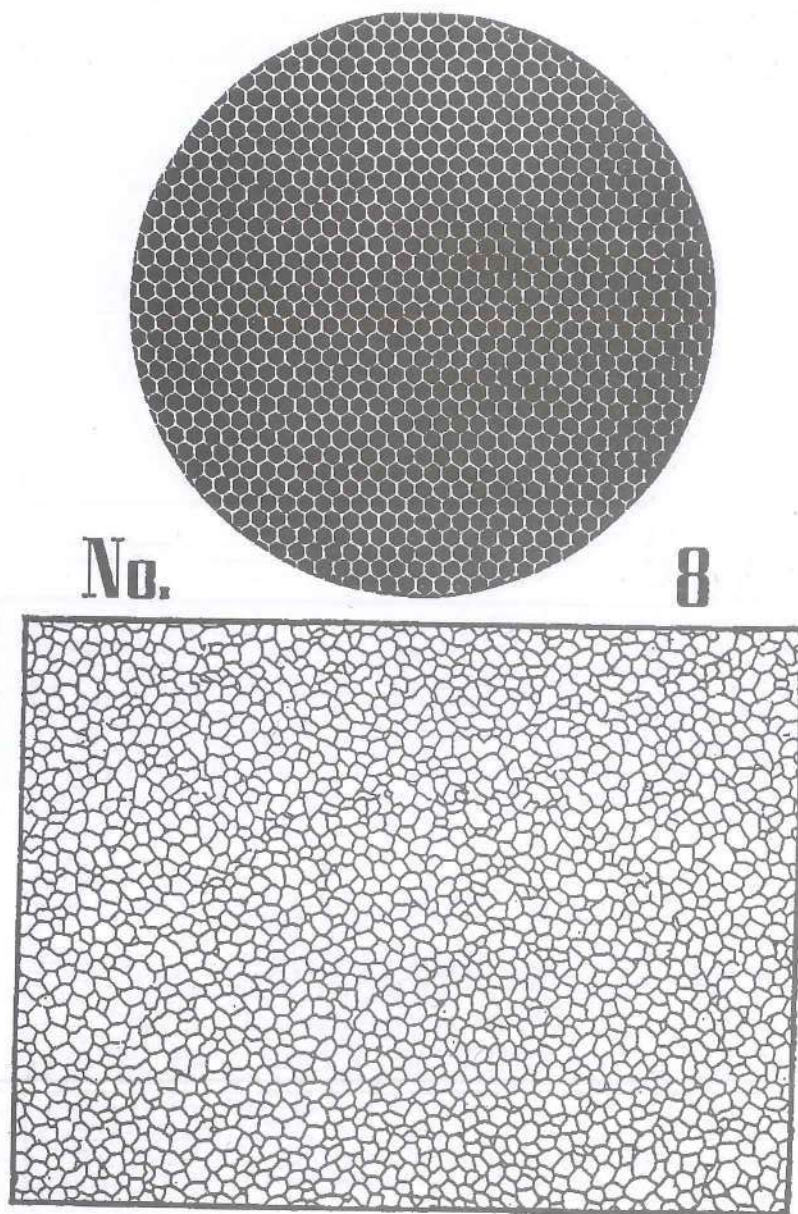


Figure 8. Upper, idealized regular shape hexagonal network for mean gran size no.8. Lower, ASTM standard grain size no.8 of irregular shape mesh. 96-192 gr/in² at 100x.

To obtain the ASTM grain size number, one must count a minimum of at least 50 grains in each of three areas, determine the number per square inch, and convert this value to the equivalent one at 100x. Then substitution in equation 1 gives N .

For grain sizes finer or coarser than the standard range of ASTM grain sizes, the magnification may be increased or decreased, still maintaining a comparison against the standard grain sizes on the chart. This enables grain sizes coarser than 1 and finer than 8 to be assessed, using the relationship:

$$N = N' + 2 (\log (m/100)) / (\log 2) \dots\dots\dots(3)$$

Where, N' is the apparent ASTM grain size at a magnification m .

For non uniform and non equiaxed grain sizes, the ASTM method is not suitable, but it is a rapid method. Being subjective, and based upon doubtful assumptions concerning grain shape and uniformity, it is at best only a semi-quantitative method.

The mean linear intercept method (Heyn method)

The mean linear intercept method (m.l.i.) is probably the widely used measurement method of grain size, particularly in metallurgical research. It is sometimes referred to as the Heyn intercept method. The m.l.i. is measured from a linear traverse by counting the number of grains which intercept a linear traverse length. If the L is the length of the intersecting line, N is the number of grains on this line, and m is the magnification thus the average diameter of the grain d can be determined by:

$$\text{m.l.i.} = d = L / (N * m) \dots\dots\dots(4)$$

When counting the number of intercepts or grains, the following should be taken into consideration:

1. If the line touches the grain boundaries, the number of grains is considered to be as $N=0.5$ (Figure 9).
2. If the line passes through a triple point of grain boundaries, the number of grains is considered to be as $N=1.5$ (Figure 10).
3. If the line starts before or goes after the grain, the number of grains is estimated as $\frac{1}{2}$ or $\frac{1}{4}$...etc (Figure 11).
4. Select a suitable microscope magnification So that the number of grains that intercept the measuring line is not less than 30 and not more than 100.
5. To get a representative grain size value, a total number of intercepts should be about 500.

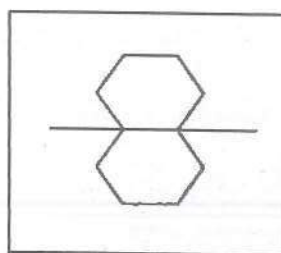


Figure 9. The line touches the Grain Boundaries

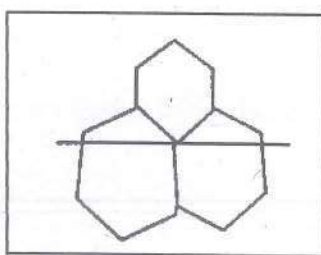


Figure 10. The line touches a triple point of Grain Boundaries

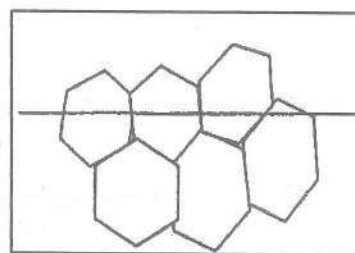


Figure 11. The line starts before or goes after the grain

It must be noted that the real grain size is greater than the measured on using the above formula because the intersection line passes through the chords of the grains and rarely through the centers of grains. Based on that, it must be appreciated that the

m.l.i. diameter (d) is less than the average diameter of grains comprising the microstructure (D) which in turn less than the maximum grain diameter because (Dm). The relationship between d, D, and Dm depend on the grain shape assumed. By assuming a reasonable shapes for real grains and obtaining a mean linear intercept of rotation:

$$D = 1.75 * d \dots\dots\dots(5)$$

Also, assuming Dm to be circumscribed sphere of rotation:

$$Dm = 1.86 * d \dots\dots\dots(6)$$

It is also possible to relate ASTM grain size numbers to values of the m.l.i. as shown in Table 1. The relationship is a geometric progression in which each value of m.l.i. is smaller than its predecessor by a factor of $1/\sqrt{2}$, d the table may be extended to larger or smaller grain sizes.

Table1. Relationship between ASTM grain size and mean linear intercept.

ASTM number	m.l.i.,d, mm
00	0.451
0	0.319
1	0.226
2	0.16
3	0.113
4	0.080
5	0.056
6	0.040
7	0.028
8	0.020
9	0.014
10	0.010
11	0.007
12	0.005

IV. The experiment

Material

Cold rolled 70 Cu % - 30% Zn brass.

Equipment

- ◆ Heat treatment furnace.
- ◆ Equipment for microscopic test preparation
- ◆ Microscope with a camera or with a scaled lense

Procedure

◆ You receive samples of cold rolled 70-30 brass. You should measure the hardness of these samples and prepare at least one for microscopic examination. Measure the grain size. The etchant consists of : ammoniacal solution of ammonium persulphate.

◆ Heat some samples of the cold worked brass to 350 °C for 3 hours then quench some of them in water and cool some in air. Measure the grain size and hardness. The etchant consists of : ammoniacal solution of ammonium persulphate.

◆ Heat some samples of the cold worked brass to 650 °C for 1/2 hours then quench some of them in water and cool some in air. Measure the grain size and hardness. The etchant consists of ammoniacal solution of ammonium persulphate.

◆ Heat some samples of the cold worked brass to 750 °C for 3 hours then quench some of them in water and cool some in air. Measure the grain size and hardness. The etchant consists of acid ferric chloride solution

◆ Heat some samples of the cold worked brass to 850 °C for 3 hours then quench some of them in water and cool some in air. Measure the grain size and hardness.

◆ Write a report discussing your observations. The etchant consists of : acid ferric chloride solution.

Student Notes

Plastic Deformation, Recrystallization, and Grain Size Calculation

<i>Name</i>		<i>Section</i>	
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Results:

The photograph of the microstructure of the cold worked specimen.

The photograph of the microstructure of the cold worked specimen heated for $\frac{1}{2}$ hour at 650 °C.

The photograph of the microstructure of the cold worked specimen heated for 3 hours at 750 °C.



The photograph of the microstructure of the cold worked specimen heated for 3 hours at 850 °C.

Grain size with different heat treatments

Grain size	Temperature	Time
	350	3
	650	1/2
	750	3
	850	3
	Room	As received

Questins:

Why is the grain size altered ?

Did you observe a grain growth ? Why?

Section II. Mass Transfer

EXPERIMENT No.5

The Surface Hardening by Carburizing and Heat treatment after carburizing

I. Purpose

To provide the student with knowledge about surface or case hardening of low carbon steel by carburising and studying the mechanical properties after using this process.

II. Background

Review the section in your materials science textbook covering Atom movement in materials.

III. Introduction

The service conditions of many steel components such as cams, gears and shafts make it necessary for them to possess both hard, wear resistant surface and at the same time, tough, shock resistant cores. The situation can be best met by employing a low carbon steel with suitable core properties. In this case carbon or nitrogen penetrates to a regulated depth to produce a potentially hard surface skin as in the principal surface hardening processes of carburizing and nitriding (see Fig 4 in experiment no. 2). Carburizing may be in solid media, in liquid media, or gas carburizing. In this experiment the carburizing in solid media (pack-carburizing) will be of concern.

Carburizing in solid media (pack-carburizing) involves packing the work into heat resisting steel (25Cr; 20Ni) boxes along with the carburizing material so that a space of approximately 50 mm exists between the components. The boxes then heated slowly to carburizing temperature and maintained at that temperature for up to a time

according to the depth of case required. Figure 1 indicates the relationship which exists between depth of case, carburising temperature and time of treatment.

Although a solid carbon-rich medium is packed around the work the actual carburising process depends upon the presence of gas carbon monoxide, CO, to carry carbon to the surface of the work pieces. Oxygen in entrapped air forms this carbon monoxide :



At the surface of the work piece this carbon monoxide releases carbon atoms:



The carbon atoms so released are dissolved interstitially at the surface of the steel.

As indicated above, charcoal alone could be used as the carburising medium in practice the rate of carburisation is increased by adding an 'energiser', usually 10-15 % barium carbonate. This dissociates at the carburising temperature :



And the carbon dioxide thus produced reacts with charcoal to form carbon monoxide :



The above reactions reversible and as the temperature is increased, pressure remaining constant, the proportion of carbon monoxide increases and so therefore does the rate

of carburizing (fig.1) In commercially produced carburizing media charcoal may be replaced by other carbon rich substances such as petroleum coke.

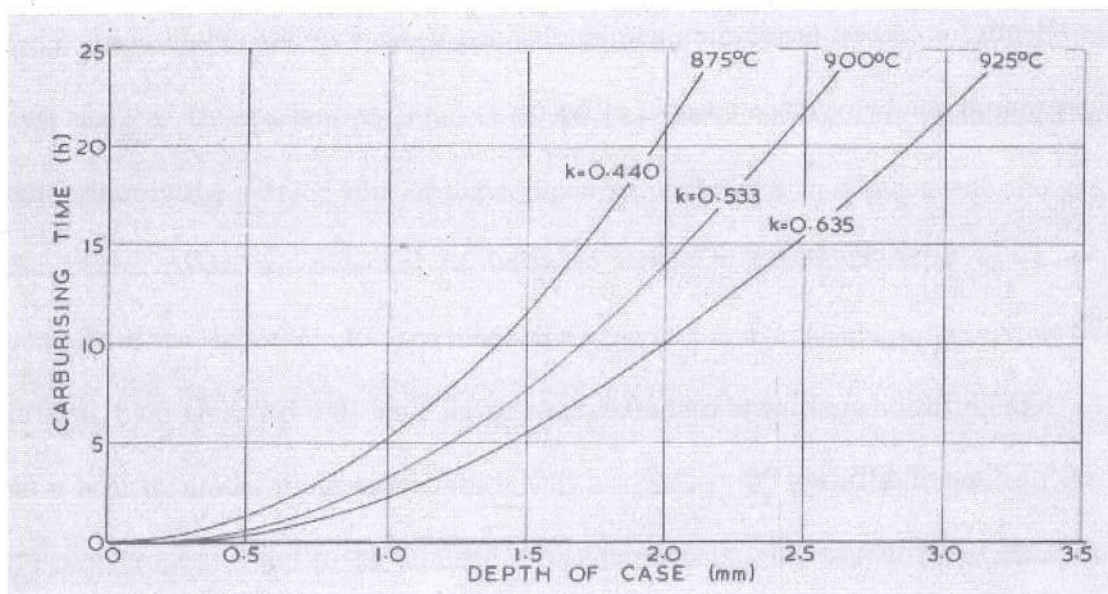


Figure 1. The relationship between time and temperature of carburizing and the depth of case produced (Higgins, 1993).

If it is necessary to prevent any areas of component from being carburized, this can be achieved by electro-plating these areas with copper at the carburizing temperature. An alternative method, which can be more conveniently applied in small scale treatment, is coat the area with a mixture of fireclay and ignited asbestos made into a paste with water. This is allowed to dry on the surface before the component is carburized. When the carburizing is complete, the components are quenched or slowly cooled in the box according to the subsequent heat treatment to be applied.

Usually Case depth is specified as total depth at which the case blends into the core of the material. There is always a transition zone in which the case blends gradually into the core and the carbon content decreases gradually. Since at least a portion of the transition zone is below 0.5% carbon, this portion achieves no appreciable hardness as

quenching. Effective case depth is considered to be that portion of case which achieves a hardness greater than 50 HRC.

Case depth for carbon penetration can be obtained using simple formula derived by Einstein from mathematical studies of diffusion:

$$\text{Case depth} = x = \sqrt{2Dt} \dots\dots\dots(1)$$

Where,

D- diffusion coefficient (cm^2/s)

t- time of diffusion (s)

It also can be written as

$$\text{Case depth} = x = k \sqrt{t} \dots\dots\dots(2)$$

Where,

$k = \sqrt{2D}$ and gives a rough estimate of time required to produce a case of a given depth.

Heat-treatment after Carburising

If carburising has been correctly carried out, the core will still be of low carbon content (0.1-0.2% carbon), whilst the case should preferably have a carbon content of no more than 0.8% C (the eutectoid composition). If the carbon content of the case is higher than this then a network of primary cementite will coincide with the grain-boundary sites of the original austenite giving rise to intercrystalline brittleness and consequent exfoliation (or peeling) of the case during service. Even if this does not occur any cracks arising from the presence of primary cementite may initiate fatigue failure. Moreover a case containing 1.0% or more carbon may be soft at the surface after quenching due to retention of austenite. After the component has been carburised

heat treatment will be necessary both to strengthen and toughen the core and to harden the case. At the same time prolonged heating in the austenitic range during carburizing order to produce an atmosphere giving the required carbon potential at the work surface. This carbon potential is in practice the carbon content maintained in equilibrium in the surface film of the component whilst it is in contact with the gas atmosphere. A carbon potential of 0.8% is usually desirable. Carrier gases are generally of the 'endothermic' type made in a generator and consisting of a mixture of nitrogen, hydrogen and will have introduced coarse grain to the whole structure, so that a heat treatment programme which will also refine the grain is desirable if the optimum properties are to be attained. For components which have been pack- or liquid-carburised to produce deep cases, a double heat-treatment is preferable to refine both core and case separately, as well as to harden the case and strengthen the core.

Refining the Core The component is first heat-treated with the object of refining the grain of the core and consequently toughening it. This is effected by heating it to just above its upper critical temperature (about 880°C for the core) when the coarse ferrite/pearlite structure will be replaced by fine austenite crystals. The component is then water quenched so that a fine ferrite/bainite/martensite structure is obtained in the core. The core-refining temperature of 880°C is, however, still high above the upper critical temperature for the case, so that, at the quenching temperature, the case may consist of large austenite grains. On quenching these will result in the formation of coarse brittle martensite. Further treatment of the case is therefore necessary.

Refining the Case The component is now heated to about 760°C, so that the coarse martensite of the case changes to fine-grained austenite. Quenching then gives a fine-grained martensite in the case.

At the same time the martensite produced in the core by the initial quench will be tempered somewhat, and much will be reconverted into fine-grained austenite embedded in the ferrite matrix (point C in Figure 2).

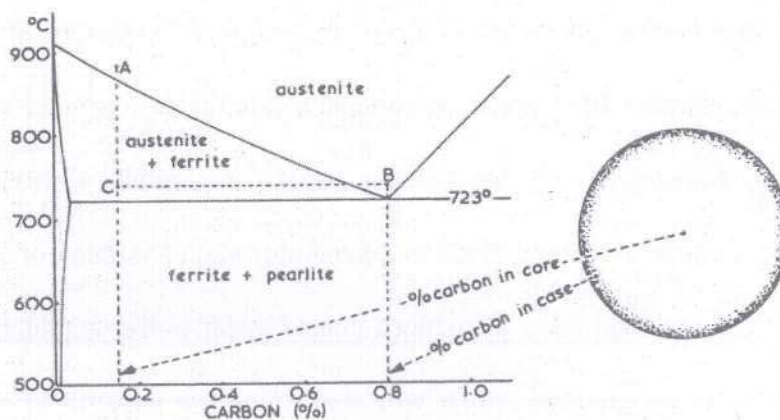


Figure 2. Heat treatment after carburizing. "A" indicates the temperature of treatment for the core and "B" the temperature of treatment of the case.(Higgins,, 1993)

The second quench will produce a structure in the core consisting of martensite particles embedded in a matrix of ferrite grains surrounded by bainite. The amount of martensite in the core is reduced if the component is heated quickly through the range 650-760°C and then quenched without soaking. This produces a core structure consisting largely of ferrite and bainite, and having increased toughness and shock-resistance. Finally, the component is tempered at between 160°C and 220°C to relieve any quenching strains present in the case.

The above comprehensive heat-treatment may be regarded as the counsel of perfection and would be that applied to important components in which cases of considerable depth had been produced and in which the necessarily prolonged carburising cycle had given rise to coarse grain. The core-refining process would also cause some reduction in the high carbon content at the skin to a more acceptable value nearer 0.8%, thus reducing the chance of primary cementite networks at the surface.

For thinner cases and lower quality work generally, modified heat-treatment is prevalent.

When thin cases (below 0.5 mm) are involved the component may be quenched direct from the carburising process with obvious advantages economically. The shorter carburising time will not have produced grain growth to the same extent as more prolonged treatment. With cases of intermediate thickness (0.5-1.25 mm) a component may be cooled slowly from the carburising temperature and given a single treatment by reheating to 820°C as a compromise between the upper and lower critical temperatures (for the core), water-quenched and finally tempered at between 160°C and 220°C. The case will then be hard though slightly coarse-grained whilst the core will consist of a ferrite/bainite/martensite structure. The ferrite will be rather coarse-grained, remaining from the original carburising and will not be completely redissolved by heating to 820°C. Hence the core will not attain maximum toughness.

IV. The Experiment

Material

- ◆ Specimens of 0.15 % C steel.
- ◆ Stainless steel box containing solid carbon powder.
- ◆ Charcoal

Equipment

- ◆ Heating furnaces
- ◆ Timing equipment

Procedure

- ◆ Put the specimen of steel in the box containing solid carbon powder.
- ◆ Put the specimen into the heating furnace.
- ◆ Switch on the furnace and let the temperature raise gradually till 950 °C.

Leave the specimen at this temperature for two hours.

- ◆ Switch off the furnace. Take out the box containing the specimen then take out the specimen from the box and put it into another furnace at temperature 780 C. Leave the specimen for 30 min. then cool it in water or oil.

- ◆ Cut the specimen into two halves.
- ◆ Prepare one half as for microscopic examination.
- ◆ Measure the thickness of the hardened layer by measuring hardness from surface to core.
- ◆ Write the report.

Student Notes

The Surface Hardening By Carburising

<i>Name</i>		<i>Section</i>	
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Results:

The micro photograph of the carburised specimen showing the depth of case hardening.

Questions:

What is the significance of surface hardening?

How temperature and time affect the depth of case produced by surface hardening?

Sectin III. Mechanical Properties of materials

EXPERIMENT No.6

Tensile Testing

I. Purpose:

To introduce the student to the method of determining the mechanical characteristics of material such as Young's modulus, Yield strength, and Ultimate strength ..etc and to introduce him to the engineering concepts related to that method such as Engineering stress, Engineering strain, True stress, and True strain.

II. Background:

Review the sections in your materials science textbook covering the mechanical properties of materials.

III. Introduction:

Deformation can be elastic or plastic. Elastic deformation occurs as long as when the applied load is released, the piece returns to its original shape (Figure1). But plastic deformation is a permanent or noncoverable deformation of the material (Fugure2). Some materials exhibit non-linear properties in the elastic region (figure3). In this case two quantities are usually employed to denote its stiffness or modulus.

The most common material property information, needed by the engineer, is contained in each material's stress-strain curve. Actual stress-strain curves, such as those of the typical materials, show that elastic deformation may only be a small part of the total data set. The general form of stress-strain curves is shown in figure4.

The main relationships to determine the mechanical characteristics from curves can be summarized as follows:

1. Engineering stress

$$\sigma = P/A_0 \dots\dots\dots(1)$$

Engineering strain

$$\varepsilon = \delta/l_0 = (l_f - l_0)/l_0 \dots\dots\dots(2)$$

2. Young's modulus or stiffness

$$E = \sigma/\varepsilon \dots\dots\dots(3)$$

for nonlinear elastic behavior

$$E_{sec} = d\sigma/d\varepsilon \dots\dots\dots(4)$$

3. Modulus of resilience

$$u = \sigma_{yield}^2 / 2E \dots\dots\dots(5)$$

4. Modulus of toughness $u_T = \int_0^{\varepsilon_T} \sigma d\varepsilon \dots\dots\dots(6)$

5. True stress

$$\sigma_T = P/A \dots\dots\dots(7)$$

True strain

$$\varepsilon_T = \ln(\delta/l_0) = \ln[(l_f - l_0)/l_0] \dots\dots\dots(8)$$

6. The relationship between Engineering (nominal) stress and true stress:

$$\sigma_T = \sigma(1 + \varepsilon) \dots\dots\dots(9)$$

7. The relationship between Engineering (nominal) strain and true strain:

$$\varepsilon_T = \ln(1 + \varepsilon) \dots\dots\dots(10)$$

8. Sometimes the curve after yield strength can be approximated by determining the constants of equation (11):

$$\sigma_T = k(\varepsilon_T)^n \dots\dots\dots(11)$$

9. Percent elongation $EL\% = [(l_f - l_0)/l_0] * 100\% \dots\dots\dots(12)$

10. Percent reduction in area $AR\% = [(-A_f + A_0)/A_0] * 100\% \dots\dots\dots(13)$

Where:

σ - Engineering stress

σ_T - True stress

σ_{yield} - Yield stress

ε - Engineering strain

ε_T - True strain

ε_r - Strain at rupture

A_0 - Initial area

A - Instantaneous area

E - Young's modulus

E_{sec} - Secant modulus

δ - Change in length or difference between final (l_f) and initial length (l_0)

u - Modulus of resilience

u_T - Modulus of toughness

n - Strain hardening exponent

k - Strength coefficient

IV. The Experiment

Material

- ◆ Standard tensile test specimen made of steel or aluminum as shown in Figure 5.

Equipment

- ◆ Computerized Universal Testing Machine (UTM). See Figure 6.

Procedure

- ◆ Prepare the standard specimen.
- ◆ Prepare the UTM and assemble the suitable jaws of the machine
- ◆ Set the suitable program for the intended work
- ◆ Choose the form of the output data
- ◆ Run the machine through the computer connected to it
- ◆ Get the data from the machine which is in load-displacement form
- ◆ Try to calculate the engineering stress and engineering strain
- ◆ Draw the engineering stress - engineering strain curve
- ◆ Try to calculate the True stress and True strain.
- ◆ Draw the True stress and True strain curve

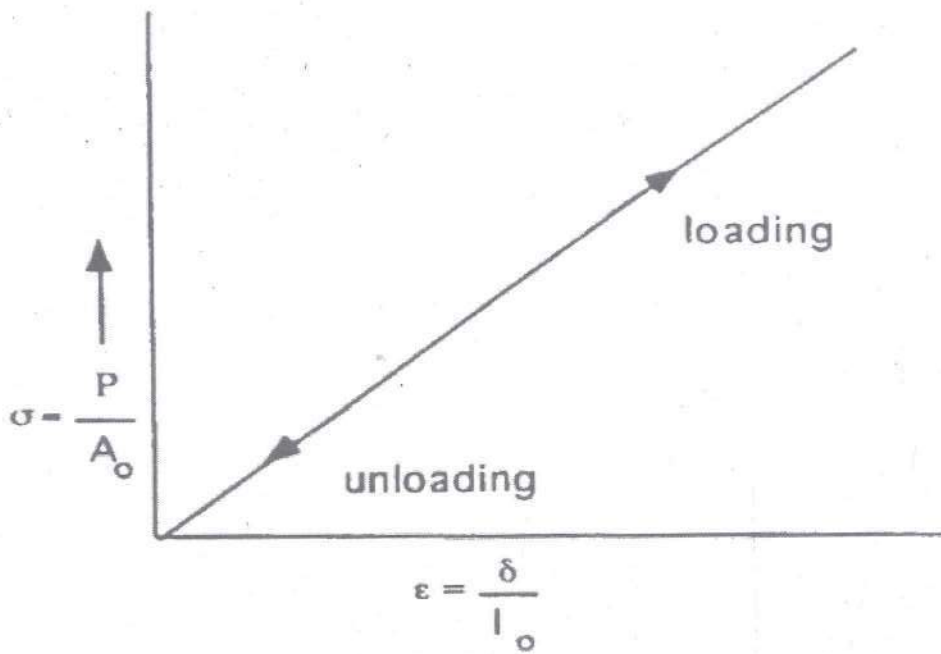


Figure1. Stress-strain curve shows the elastic behavior of the material.

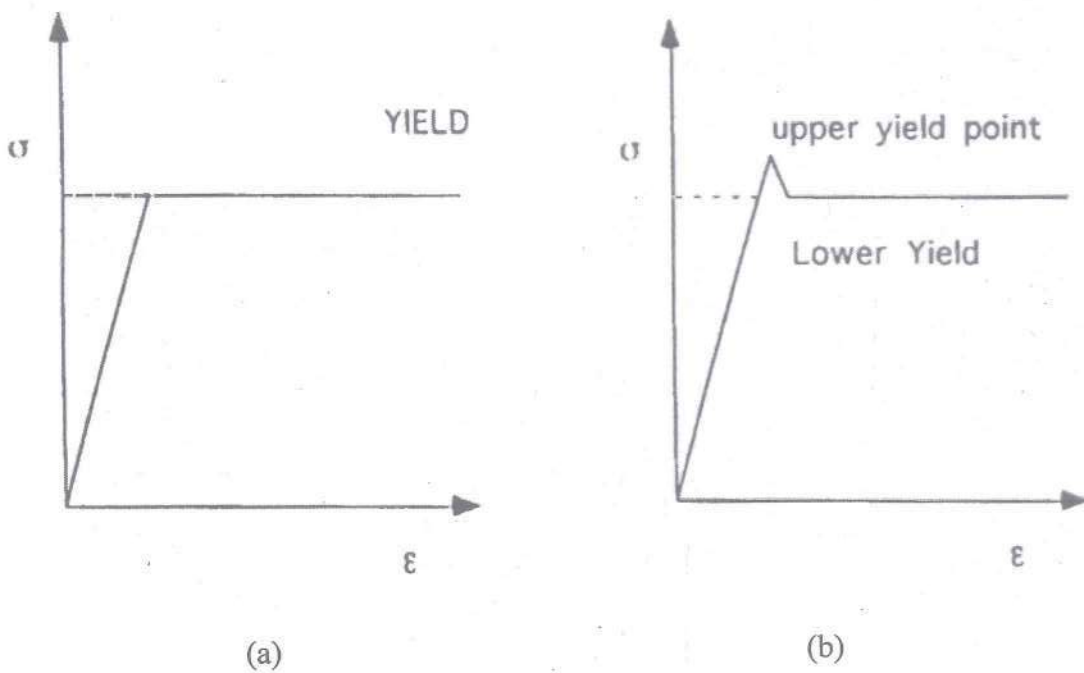


Figure2. Stress-strain curve shows two types of the plastic behavior of the material.

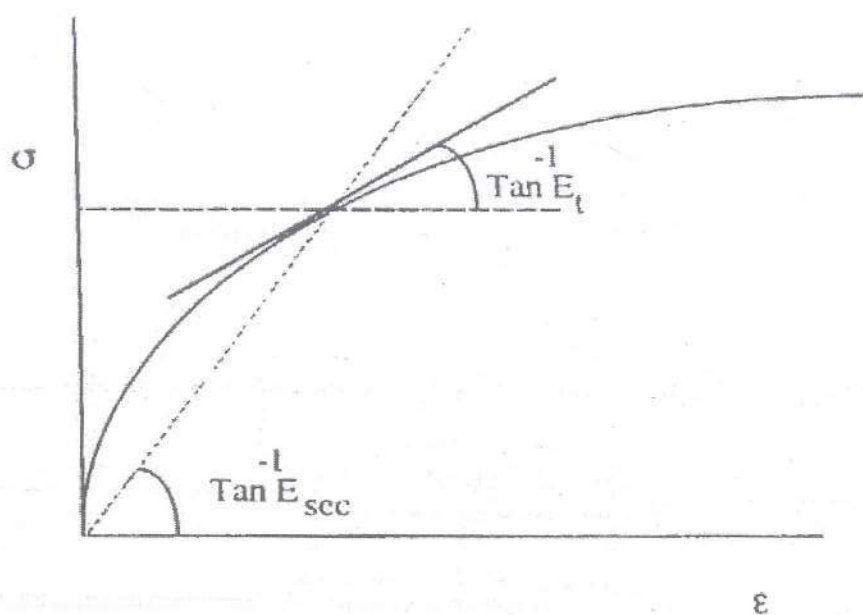


Figure3. Non linear elastic behavior of material.

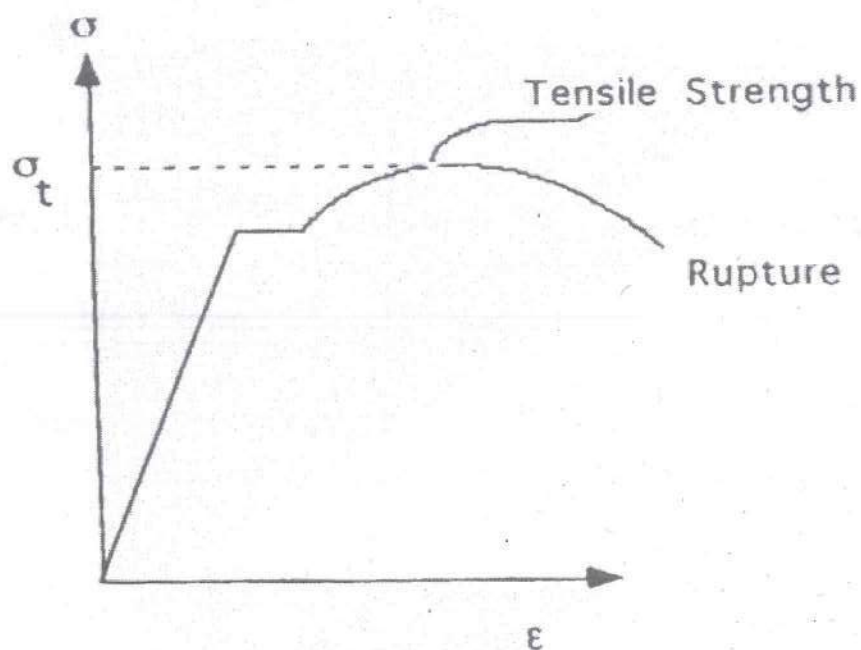
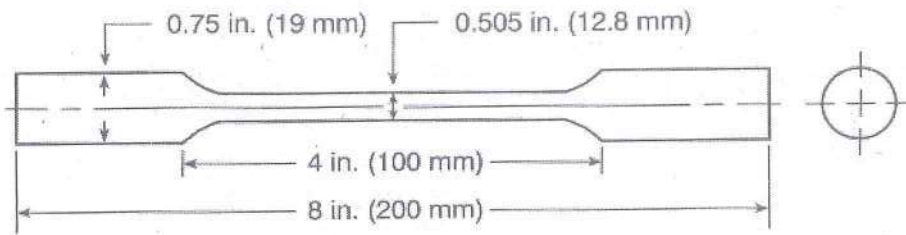
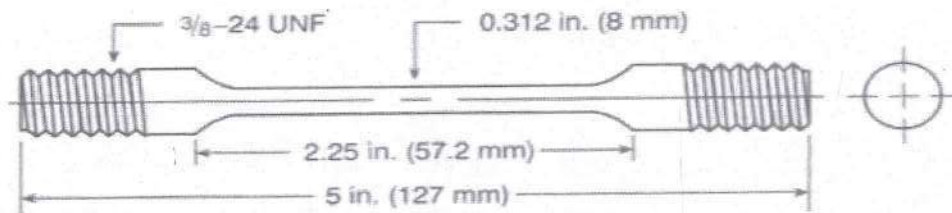


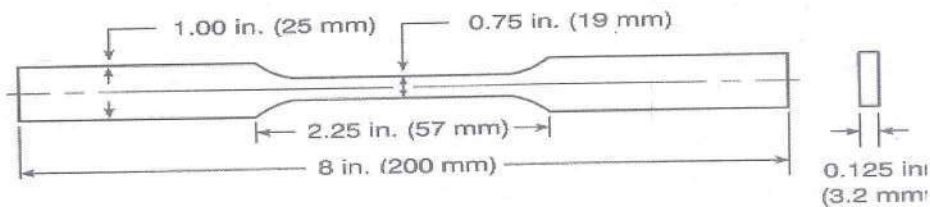
Figure4. General form of the stress-strain curve.



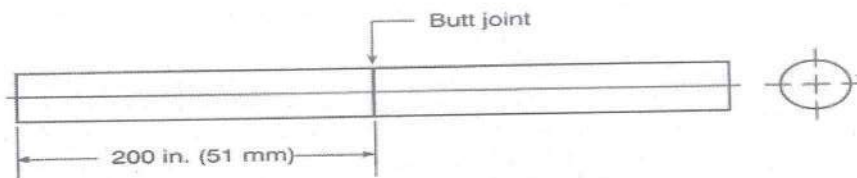
a) Smooth-end round tensile test specimen



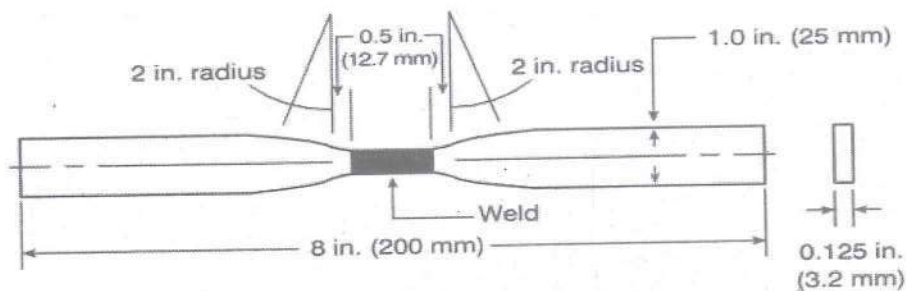
b) Threaded end tensile test specimen



c) Flat tensile test specimen



d) Brazed or silver-soldered tensile test specimen



e) Weld tensile test specimen

Figure5. Standard tensile testing specimens (Larry Horath, 2001)

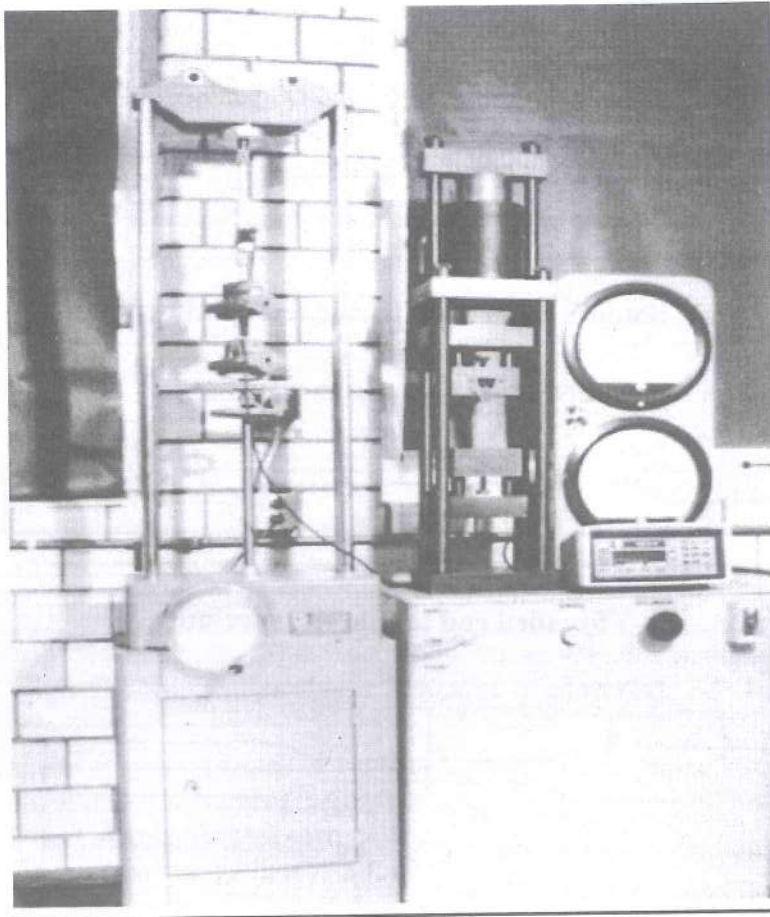


Figure 6. Universal tensile testing machine (Larry Horath, 2001)

Student Notes

Engineering stress-engineering strain plot

True stress-true strain plot

The main mechanical properties extracted from the stress-strain curves

<i>Property</i>	<i>Its value</i>
Young's modulus	
Yield stress	
Ultimate Tensile Strength	
True stress at rupture	
Engineering stress at rupture	
Modulus of resilience	
Modulus of toughness	
Yield point at 0.2% strain	
% elongation at rupture	
% reduction of area at rupture	
Strength coefficient	
Strain hardening exponent	

Questions:

Compare the shapes of engineering stress strain curves and those of true stress –true strain.

What are the probable errors in this test? Discuss briefly how they affect the accuracy of the Young's modulus and yield point determination?

EXPERIMENT No 7

Creep Test

I. Purpose

The student should recognize and define the time and temperature dependence in creep deformation and specify the conditions under which it occurs. He also should construct a results curve in terms of strain versus time at a given temperature and load and describe the three regions of the creep curve.

II. Background

Review the sections in your materials science textbook covering the subject: Creep and creep testing.

III. Introduction

The **deformation** at elevated temperature under constant **load** is called **creep**. It depends on the wire material and the temperature of the room. The response of the material to an applied load will depend on the temperature and the amount of weight we use. When we talk about the temperature of a material, we are actually more interested in the **absolute temperature** (T_{abs}) of the material compared to the material's **absolute melting temperature** (T_{MPabs}). We call this the **homologous temperature** and can designate it by the ratio (T_{abs}/T_{MPabs}).

When we hang a weight on a material in a wire at a low homologous temperature ($T_{abs}/T_{MPabs} < 0.4$), the wire elongates. If that weight is relatively small, the wire stretches just a little. As soon as we remove it, the wire returns to its original length. This is an example of a reversible deformation, or an **elastic deformation**, like a spring. When we hang a medium weight on the wire at low T_{abs}/T_{MPabs} , it stretches more than before. When we remove the weight, it does not return to its original

length. This permanent stretching of the wire is known as **plastic deformation**. If the weight is too heavy, the wire fractures.

If we raise the temperature so that $T_{abs}/T_{MPabs} > 0.4$, and hang a weight on it that does not noticeably stretch the wire, it may initially appear that we have only elastically deformed the wire. At temperatures above about $T_{abs}/T_{MPabs} = 0.4$, however, atoms begin to move about in the solid at significant rates. This atomic movement can lead to time dependent stretching of the wire under a load even when the weight is very small. If we observe the length of the wire over a long time (hours, days, or weeks), we notice that the wire very slowly elongating. This time dependent elongation of the wire is called **creep**.

Creep is an important consideration in any application where a component must support a load at temperatures where $T_{abs}/T_{MPabs} > 0.4$. A jet engine is one good example where a material operates at very high temperatures (about 1100 K, or Kelvin degrees). Because the engine temperatures are so high, the **alloys** used for the turbine blades operate at temperatures very close to their melting temperatures. They are called **superalloys**. In order to demonstrate creep in alloys without using very high temperatures, we can observe creep in low melting point alloys at temperatures near room temperature (about 300 K). In design, we seek materials that will carry the design loads without failure for the design life at the service temperature. Creep is an important consideration in design in three types of high temperature applications:

1. Displacement-limited applications in which precise dimensions or small clearances must be maintained such as in turbine rotors in jet engines)
2. Rupture-limited applications in which precise dimensions are not essential but fracture must be avoided such as in high-pressure steam tubes and pipes.

3. Stress-relaxation-limited applications in which an initial tension relaxes with time such as in suspended cables and tightened bolts.

In these types of applications, design engineers must consider creep deformation and its dependence on time and temperature. Many mechanical systems and components like turbines, steam boilers, and reactors operate at high temperatures and creep properties for

the materials used must be determined.

The main objective in a creep test is to measure how a given metal or an alloy will perform under constant load, at elevated temperatures. In a creep test, a tensile specimen (with similar dimensions as a tensile test specimen) is subjected to a constant load inside a furnace where the temperature is maintained constant. Figure 2 illustrates a simple setup for creep testing. The resulting deformation or strain is measured and plotted as a function of elapsed time. Figure 1 shows a schematic creep curve for a metal tested at constant load until rupture. Metals, polymers, and ceramics all show similar strain-time behaviors.

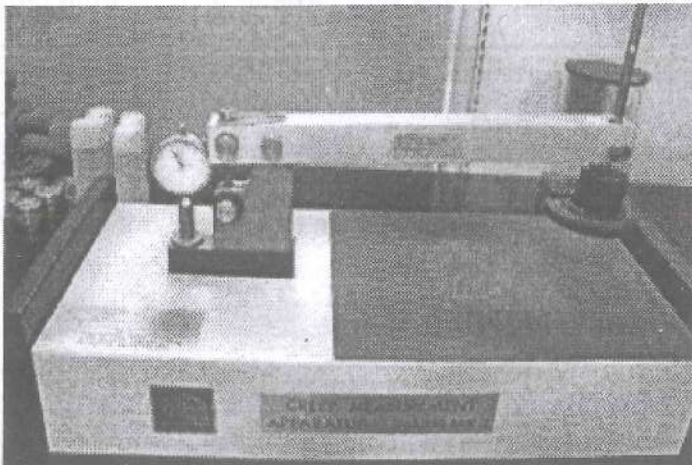


Figure 1. A creep test set up.

The creep curve in Figure 2 demonstrates three regions of strain-time behavior:

1. Primary creep where the rate of change of strain (creep rate= $\Delta s / \Delta t$) decreases with time due to strain hardening of the material.

2. Steady-State creep where the strain increases linearly with time. From design point of view, this region is the most important one for parts designed for long service life because it comprises the longest creep duration. The main creep test result is the slope of this region which is known as the steady-state creep rate ($\dot{\epsilon}$). During this stage of creep, there is a balance between strain hardening due to deformation and softening due to recovery processes similar to those occurring during the annealing of metals at elevated temperature.
3. Tertiary-creep where the strain increases rapidly until failure or rupture. The time to failure is often called the time to rupture or rupture lifetime (t_r). This parameter is an important consideration in designing against creep for parts intended for short-life applications. To determine the rupture lifetime, the creep test must be conducted to the point of failure. Such test is also known as the stress rupture test or creep rupture test.

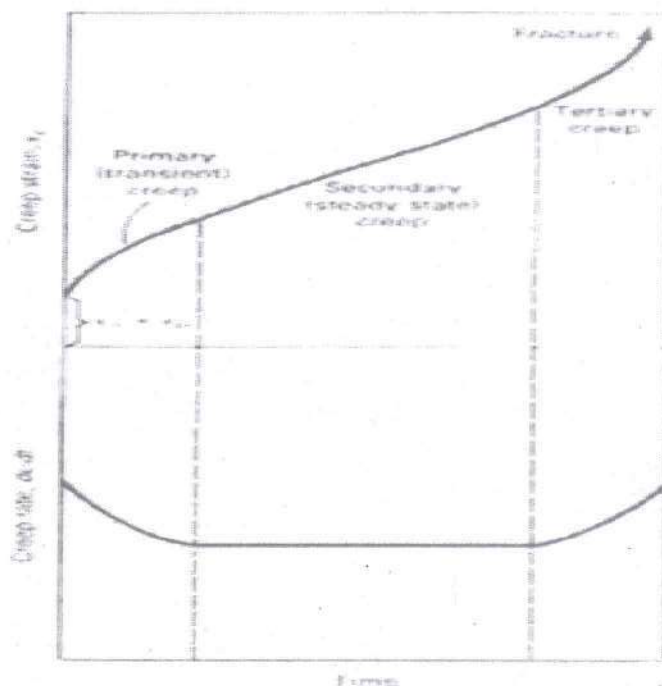


Figure 2. A creep curve

Testing either at higher stresses or higher temperatures will increase the steady-state creep rate and reduces the rupture lifetime as illustrated in Figure 3. Note that the strain is constant and independent of time for temperatures below $0.4 T_m$.

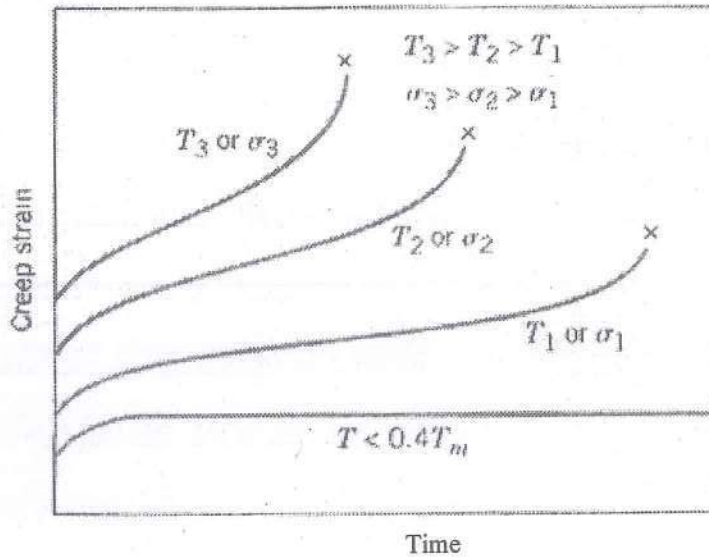


Figure 3. The creep curve at higher stresses or temperatures

IV. The Experiment

Material

To demonstrate creep behavior at room temperature, we can use wire of soldering material that contains 60 wt% Sn (tin) and 40 wt% Pb (lead). The wire Diameter = 3.175 mm. Its melting temperature = $183^{\circ}\text{C} = 456\text{ K}$ and the hanging load = 4.8 kb = 10.6 lbs.

The phase diagram for the Pb-Sn alloy system is shown in Figure , and the composition of the solder is identified by the red vertical line. One of the uses for a phase diagram is to determine the melting temperature of an alloy. The phase diagram indicates that the 60/40 solder melts at about $183^{\circ}\text{C} = 456\text{ K}$.

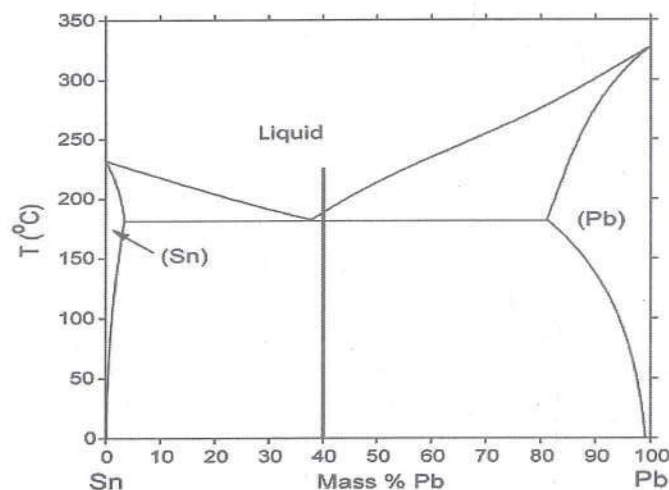


Figure .Lead-Tin Phase Diagram

Apparatus

The creep machine is shown in Figure 2.

Procedure

- To determine the homologous temperature at room temperature (25°C), the calculation is as follows:

1. Convert room temperature (25°C) to absolute temperature in Kelvins:

$$T_{abs} = 25 + 273 = 298 \text{ K} \dots \dots \dots (1)$$

2. Convert the melting temperature of the alloy (183°C) to absolute temperature in Kelvins:

$$T_{MPabs} = 183 + 273 = 456 \text{ K} \dots \dots \dots (2)$$

3. The ratio of the absolute room temperature (298 K) to the absolute melting temperature of the alloy (456 K) is the homologous temperature:

$$T_{abs}/T_{MPabs} = (298 \text{ K}) / (456 \text{ K}) = 0.653 \dots \dots \dots (3)$$

This means that room temperature corresponds to a homologous temperature of $T_{abs}/T_{MPabs} = 0.65$ for the 60/40 solder. Because the solder is at a relatively high fraction of its melting temperature, we expect to observe creep deformation in solder at room temperature.

- The load applied to the wire is determined to be 4.8 kg, which is only about one tenth of the load required to get immediate plastic deformation.
- Test temperature is 77°F and test duration is 90.8 hours = 3 days, 18 hours, 48 seconds.

- Plot of the wire length versus time during the creep test. The wire is initially 30 cm long.
- Remove any load from the arm.
- Write down the specimen cross-sectional area and its gauge length.
- Carefully mount the lead specimen in the upper and lower grips.
- Zero the dial gauge (which measures the elongation in mm).
- Gently apply the load as per the instruction of your instructor.
- Immediately record the instantaneous elongation on the dial.
- Record the elongation every 30 seconds until failure.
- Perform the test at least twice at two different loads and at room temperature (800 and 1000g).
- Repeat the tests at the same loads used above but at higher temperatures (35 °C)

Student Notes

Creep test

<i>Name</i>		<i>Section</i>	
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Results:

Plot the strain as a function of time for the test

EXPERIMENT No. 8

Hardness Testing

I. Purpose:

To determine the Hardness of the material as it is one of the required properties for some service conditions.

II. Background:

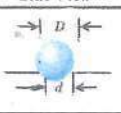
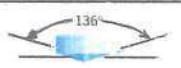
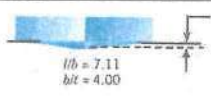
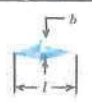
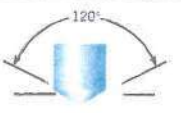
Review the sections in your materials science textbook covering the mechanical properties of materials.

III. Introduction:

The material is selected by matching its mechanical properties to the required service conditions of the component. This means that the material is valuable to the engineer if it meets the required mechanical properties. These mechanical properties are strength, hardness, toughness, fatigue resistance... etc. This experiment will be of value for engineer because it deals with the determination of the hardness of the material.

Hardness of a metal is its resistance to surface indentation under standard test conditions. It can also be considered as the resistance of material to localized plastic deformation. Three main test methods, that are used to determine hardness, are Brinell, Vickers, Microhardness (Knoop), and Rockwell tests. A summary of some important parameters of these techniques, such as indenter type, shape of indentation, calculation formula, and required testing loads, are shown in Table 1.

Tale 1. Parameters of hardness testing techniques. (Ref. H. W. Hyden et al, The structure and properties of materials, vol. III. Mechanical behavior, John Wiley and sons Inc, 1965)

Test	Indenter	Shape of Indentation		Load	Formula for Hardness Number ^a
		Side View	Top View		
Brinell	10-mm sphere of steel or tungsten carbide			P	$HB = \frac{2P}{\pi D [D - \sqrt{D^2 - d^2}]}$
Vickers microhardness	Diamond pyramid			F	$HV = 1.854P/d_1^2$
Knoop microhardness	Diamond pyramid			P	$HK = 14.2P/l^2$
Rockwell and Superficial Rockwell	Diamond cone; $\frac{1}{16}, \frac{1}{8}, \frac{1}{4}, \frac{1}{2}, 1$ in. diameter steel spheres	  	60 kg 100 kg 150 kg Rockwell 15 kg 30 kg 45 kg Superficial Rockwell		

^a For the hardness formulas given, P (the applied load) is in kg, while D, d, d_1 , and l are all in mm.

Source: Adapted from H. W. Hayden, W. G. Moffatt, and J. Wulff, *The Structure and Properties of Materials*, Vol. III, *Mechanical Behavior*. Copyright © 1965 by John Wiley & Sons, New York. Reprinted by permission of John Wiley & Sons, Inc.

Brinell hardness test

In the Brinell test an indenter consisting of a 10 mm case-hardened steel or tungsten carbide ball mounted in a suitable holder, is forced into a surface of the test-piece using a suitable load (3000 kg for hard metals, 1500 kg for intermediate hardness, and 500 kg or lower for soft materials), which is maintained for a minimum of 30 seconds for ferrous metals and 60 seconds for softer metals (Figure 1). The diameter of the circular indentation left in the surface after removal of the load is measured in two directions at right angles using low-power graduated microscope. The average diameter is taken.

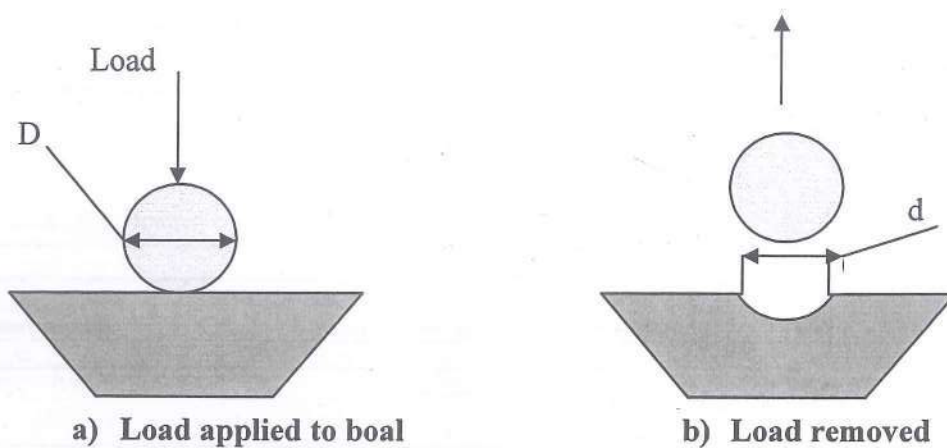


Figure 1. Brinell hardness test

The Brinell hardness number (BHN or HB) is nominally the pressure per unit area in kilograms per square millimeter. It is obtained by dividing the applied load by the area of the surface indentation, which is assumed spherical. The applied load and the diameter of indentation are entered into the following formula:

$$BHN \text{ or } (HB) = \frac{2P}{\pi D[D - \sqrt{D^2 - d^2}]} \dots\dots\dots(1)$$

Where:

D - ball diameter (mm)

d - diameter of circular indentation (mm)

P - load, which, in hardness testing, has units of kilogram-force (1kgf = 9.81 Newton)

However to avoid the tedious calculation, the Brinell hardness number may be found from tables relating hardness to the diameter of the indentation. Each table of such relationships refers to a specific load and ball diameter. In carrying out a Brinell test certain requirements must be met. For example: the depth of indentation must be not too great relative to the thickness of the test piece otherwise the table supporting the

test piece would be taking the load and not the test piece itself. For soft materials, the thickness of the test-piece should be at least 15 times the depth of the indentation while for hard materials the thickness should be at least 7 times the depth of the indentation. In general, the material's thickness should be no less than 10 times the depth of the indentation. When thinner specimens are tested, the hardness of the anvil underneath the specimen is what is really being tested.

The symbol HB is supplemented by numbers indicating the diameter of the ball used and the load applied. Thus 248 HB 10 /3000 indicates that a Brinell hardness of 248 was obtained by using a 10-mm diameter ball with a load of 3000 kgf.

The Brinell test using a hardened steel ball is not reliable for materials of hardness greater than 450 HB because of possible deformation of the indenter. The Brinell test makes a relatively large indentation, which is desirable, when it is necessary to obtain the average hardness of heterogeneous material (e.g. Grey cast iron). However, large indentations may be objectionable.

The Brinell hardness number usually falls within a range of 90 to 630, with higher numbers indicating greater hardness. The deeper the penetration, the larger the diameter of the indentation and the lower the hardness number. If the penetrator leaves an indentation with a diameter greater than 6 mm, a lighter load should be used. When the penetrator is driven into the surface of the material, a ridge develops around the indentation. This ridge tends to give a false reading on the diameter of the indentation.

On the upper end of the scale, Brinell numbers over 650 should not be trusted for two reasons:

- (1) The diameter of the indentation is so small that very slight inaccuracies in readings will greatly affect the BHN.

(2) the ball penetrator tends to flatten out and give inaccurately high diameters. Not many common materials have BHNs over 650.

As a rule of thumb, a 3000-kg load should be used for a BHN of 150 and above; a 1500-kg load, for BHNs between 75 and 300, and a 500-kg load, for materials with BHNs below 100. These figures overlap to give a reasonable latitude in testing.

The Brinell test has some limitations and disadvantages. It is a destructive test because it leaves an indentation in the material. In many cases, this renders the specimen unfit for use. In addition, many Brinell testing machines are heavy (more than 200 lb), which makes them cumbersome and unfit for field service. They also tend to be more expensive than other machines. For example, a basic Brinell testing machine with accessories and microscope generally costs more than \$5000. The test itself tends to be subjective, in that the training, experience, and attitude of the technician measuring the indentation may vary the results. This variation is generally low, but two technicians performing the same test on the same specimen may have results that vary up to an average of 10%. The necessity of calculating the BHN rather than obtaining the number from direct reading is also considered a limitation and disadvantage. The Brinell test has some advantages. Because it is older and is well established, most people are acquainted with the test, and the test results are generally accepted throughout industry. The test can be performed quickly, generally in about 2 min.

Vickers hardness test

Vickers hardness test uses a diamond indenter, in the form of a right pyramid with a square base and an angle of 136° between opposite faces, which is forced into the prepared surface of the test material under a selected load (Figure 2). The diamond

produces a square indentation and the average is taken of the diagonal length, which are measured by means of a microscope with a variable slit built into the eyepiece.

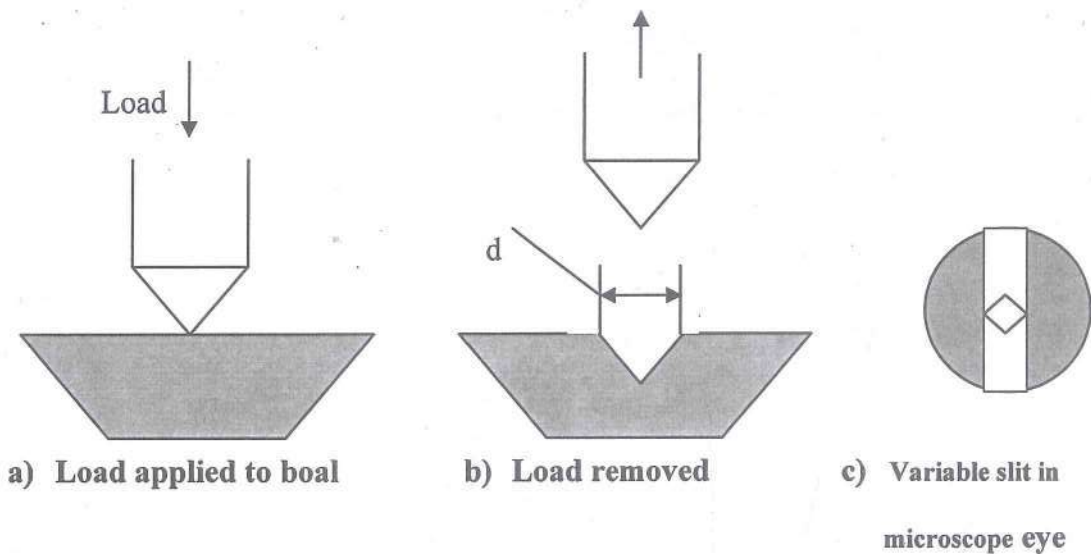


Figure 2. Vickers hardness test

The width of the slit is adjusted so that its edges coincide with the corners of the indentation and the diagonal length is then obtained from a revolution counter geared to the movement of the slit

The ocular reading obtained is converted to a Vickers hardness number HV (or sometimes called Diamond Pyramid Hardness (DPH)) by referring to the tables thus obviating tedious calculation. However as with the Brinell test the hardness is defined as:

$$HV \text{ (or DPH)} = 1.854 P/d^2(2)$$

Where

P - load in kgf

d - diagonal length in mm

Loads between 5 and 120 kg in 5 kg increments can be used depending on the hardness and thickness of the material being tested. The rate and duration of loading (15 seconds) are controlled automatically by a piston working in an oil dashpot.

The symbol HV is supplemented by a number indicating the load used in the test; thus 650 HV/30 represents a Vickers hardness number of 650 obtained with a load of 30 kgf. Because the indentation made by the diamond is much smaller than that made in the Brinell test, a smoother finish is required on the test material for Vickers hardness testing. This means that the Vickers test is more suitable than the Brinell test for testing finished components. The sample is prepared as for microstructural test in experiment No.3.

The Vickers test provides a suitable hardness scale for material ranging from the very soft to very hard material up to a hardness number of about 300 the Brinell and Vickers hardness values are nearly the same. But the higher values the Brinell results are the lower due to distortion of the ball indenter.

As stated previously the Brinell hardness number is not reliable above 450 HB. The hardness of the hardened steel ball indenter used in the Brinell must be not less than 850 HB.

The Vickers test is used primarily in research applications. One advantage claimed by some Vickers machine operators is in the measurement of the indentation: A much more accurate measure can be made of the diagonal of a square than can be made on a circle, where the measurement is made between two tangents to the circle. It is a fairly rapid method and can be used on metal as thin as 0.006 in. It is claimed to be accurate for hardness as high as 1300 (about 850 Brinell). The indenter does not exhibit the tendency to flatten out as much as with the Brinell test.

Disadvantages are that the Vickers test is a destructive test. The test is much slower than either the Brinell or the Rockwell tests. To use the Vickers test, the surface of the specimen must be polished, which takes considerable time. Although the Vickers test is more accurate than either the Brinell or Rockwell tests, the cost of the equipment is much higher, although the results are as widely accepted.

Knoop hardness test and microhardness tester

The Knoop indenter is made of diamond and is ground so that it makes a diamond shaped indentation, the ratio of the long to the short diagonals being 7:1. The Tukon tester, with which the Knoop indenter is used, can apply loads of 25 to 3600 g. It is fully automatic in making the indentation. The operator places the specimen under a microscope and selects an area for testing. The test is conducted, and the indentation is measured under a microscope. The Knoop hardness number is the ratio of the applied load (in kg) to the unrecovered projected area (in mm²):

$$KHN = 1.43 * PL/d^2 \dots\dots\dots(3)$$

where:

KHN: Knoop hardness number (in km/mm²)

P : load (in kg)

d : length of longest diagonal (in mm)

The Knoop hardness number is between 60 and 1000. It is a measure of the resistance of the material's surface to penetration, or a measure of the surface plasticity of the material. The Tukon-Knoop device and the similar Wilson-Knoop device are useful for hardness testing on small parts or for testing hardness over a small area. Although

the Tukon tester is often supplied with a Knoop indenter, it can also be adapted for use with a Vickers 136° diamond-pyramid indenter. Figure 3 shows the Knoop indenter.

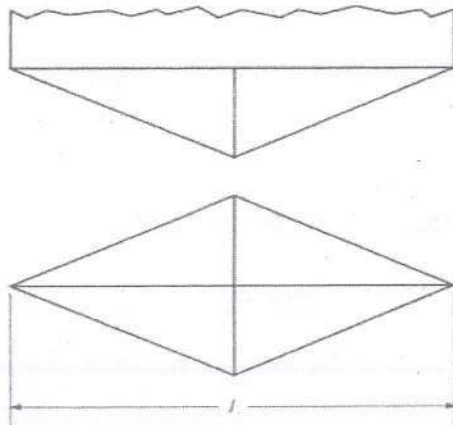


Figure 3. Knoop Indenter

Rockwell hardness test

The Rockwell hardness test is made by applying two loads to a specimen and measuring the difference in depth of penetration in the specimen between the minor load and the major load. The minor load is used on the standard Rockwell tester to eliminate errors that could be caused by specimen surface irregularities. The minor load is 10 kilograms of force (kgf) when used with 60, 100, or 150 kgf major load and 3 kgf on the superficial tests where the major loads are 15, 30, and 45 kgf. The major load is applied after the minor load has seated the indenter firmly in the work. The Rockwell hardness reading is based on the additional depth to which the penetrator is forced by the major load (Figure 4). The depth of penetration is indicated on the dial when the major load is removed. The amount of penetration decreases as the hardness of the specimen increases. Generally, the harder the material is, the greater its tensile strength will be, that is, its ability to resist deformation and rupture when a load is

applied (see Table 1 in Appendix E). The Rockwell C scale is used for hard metals such as heat-treated steels. A file, for example, may measure RC 65, a steel spring about RC 45, and a good knife between RC 52 and 58. Since the C scale below RC 15 is somewhat unreliable, the B scale should be used for these softer metals. Soft iron may read from 0 to 10 on the C scale but would read from 80 to 90 on the B scale.

There are two basic types of penetrators used on the Rockwell tester (Figure 5). One is a sphero-conical diamond called a Brale that is used only for hard materials, that is, for materials over B-100 such as hardened steel, nitrided steel, and hard cast irons. When the C Brale diamond penetrator is used, the recorded readings should be prefixed by the letter "C." The major load used is 150 kgf. The C scale is not used to test extremely hard materials such as cemented carbides or shallow case-hardened steels and thin steel. An A Brale penetrator is used in these cases and the A scale is used with 60 kgf major load.

The second type of penetrator is a $1/16$ -in. diameter steel ball that is used for testing material in the range of B-100 to B-0, including such relatively soft materials as brass, bronze, and soft steel. If the ball penetrator is used on materials harder than B-100, there is a danger of flattening the ball. Ball penetrators for use on very soft bearing metals are available in sizes of $1/2$, $1/4$, and $1/8$ in. (Table 2). Figure 6 points out the parts used in the testing operation on the Rockwell hardness tester.

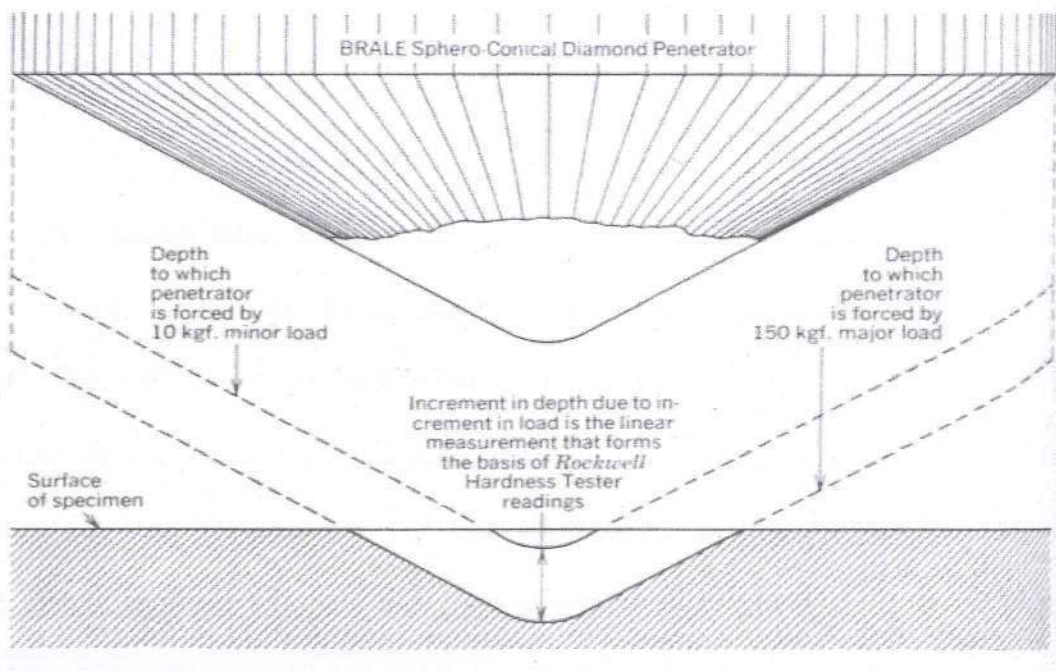


Figure 4. Schematic showing minor and major loads being applied. (Wilson Instrument Division of Acco)

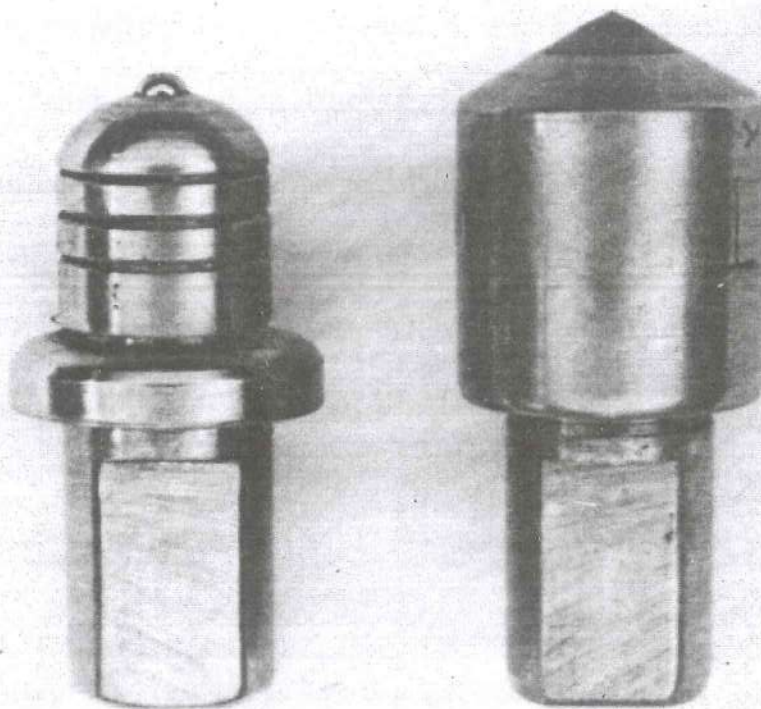
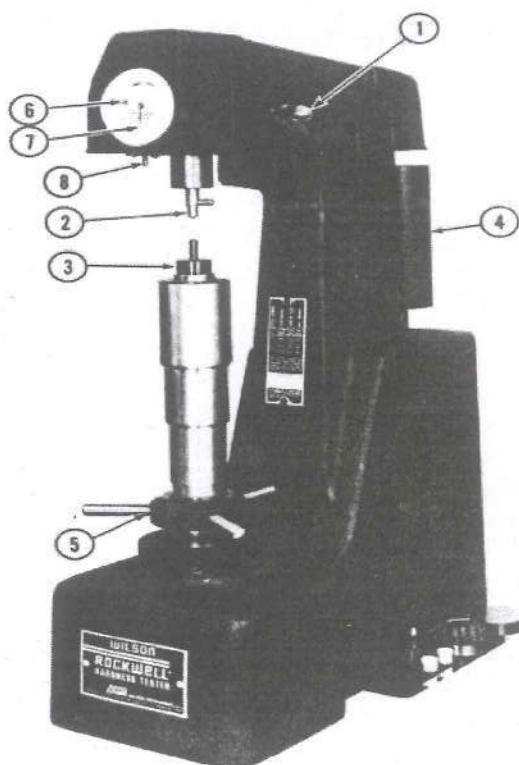


Figure 5. Braille and Ball. (Braille is a registered trade mark of American Chain & Cable company, Inc.)

Table 2. Penetrator and load selection. (Wilson Instruction Manual, American Chain and Cable Company, Inc., 1973)

Scale Symbol	Penetrator	Major Load (kgf)	Dial Figures	Typical Applications of Scales
B	1/16 in. ball	100	Red	Copper alloys, soft steels, aluminum alloys, malleable iron, etc.
C	Brale	150	Black	Steel, hard cast irons, pearlitic malleable iron, titanium, deep case-hardened steel, and other materials harder than B-100
A	Brale	60	Black	Cemented carbides, thin steel, and shallow case-hardened steel
D	Brale	100	Black	Thin steel, medium case-hardened steel, and pearlite malleable iron
E	1/8 in. ball	100	Red	Cast iron, aluminum and magnesium alloys, and bearing metals
F	1/16 in. ball	60	Red	Annealed copper alloys, thin, soft sheet metals
G	1/16 in. ball	150	Red	Phosphor bronze, beryllium copper, malleable irons. Upper limit G-92 to avoid possible flattening of ball
H	1/8 in. ball	60	Red	Aluminum, zinc, lead
K	1/8 in. ball	150	Red	
L	1/4 in. ball	60	Red	
M	1/4 in. ball	100	Red	Bearing metals and other very soft or thin materials. Use the smallest ball and heaviest load that does not give an anvil effect.
P	1/4 in. ball	150	Red	
R	1/2 in. ball	60	Red	
S	1/2 in. ball	100	Red	
V	1/2 in. ball	150	Red	



1. Crank handle.
2. Penetrator.
3. Anvil.
4. Weights.
5. Capstan handwheel.
6. Small pointer.
7. Larger pointer.
8. Lever for setting the bezel.

Figure 6. Rockwell hardness tester. (Wilson Instrument Division of Acco)

Superficial Testing

After testing sheet metal, examine the underside of the sheet. If the impression of the penetrator can be seen, then the reading is in error and the superficial test should be used. If the impression can still be seen after the superficial test, then a lighter load should be used. A minor load of 3 kgf and a major load of 30 kgf is recommended for most superficial testing. Superficial testing is also used for case-hardened and nitrided steel having a very thin case. A Brale marked N is needed for superficial testing, as A and C Brales are not suitable. Recorded readings should be prefixed by the major load and the letter N when using the brale for superficial testing, for example, 30N78. When using the 1/16 -in ball penetrator, the same as that used for the B, F, and G hardness scales, the readings should always be prefixed by the major load and the letter T, for example, 30T85. The 1/16 -in ball penetrator, however, should not be used on material harder than 30T82. Other superficial scales, such as W, X, and Y, should also be prefixed with the major load when recording hardness. See Table 3 for superficial test penetrator selection.

Table 3. Superficial tester load and penetrator selection. (Wilson Instrument Division of Acco)

Scale Symbol	Penetrator	Load (kgf)
15N	Brale	15
30N	Brale	30
45N	Brale	45
15T	1/16 in. ball	15
30T	1/16 in. ball	30
45T	1/16 in. ball	45
15W	1/8 in. ball	15
30W	1/8 in. ball	30
45W	1/8 in. ball	45
15X	1/4 in. ball	15
30X	1/4 in. ball	30
45X	1/4 in. ball	45
15Y	1/2 in. ball	15
30Y	1/2 in. ball	30
45Y	1/2 in. ball	45

The basic anvils used for Rockwell testing are shown in Figure 7. Flat anvils are used for specimens with flat surfaces and V-type anvils for round specimens. A spot anvil is used when the tester is being checked on a Rockwell test block. The spot anvil should not be used for checking cylindrical surfaces. The diamond spot anvil (Figure 8) is similar to the spot anvil, but it has a diamond set into the spot. The diamond is ground and polished to a flat surface. This anvil is used only with the superficial tester, and then only in conjunction with the steel ball penetrator for testing soft metal.

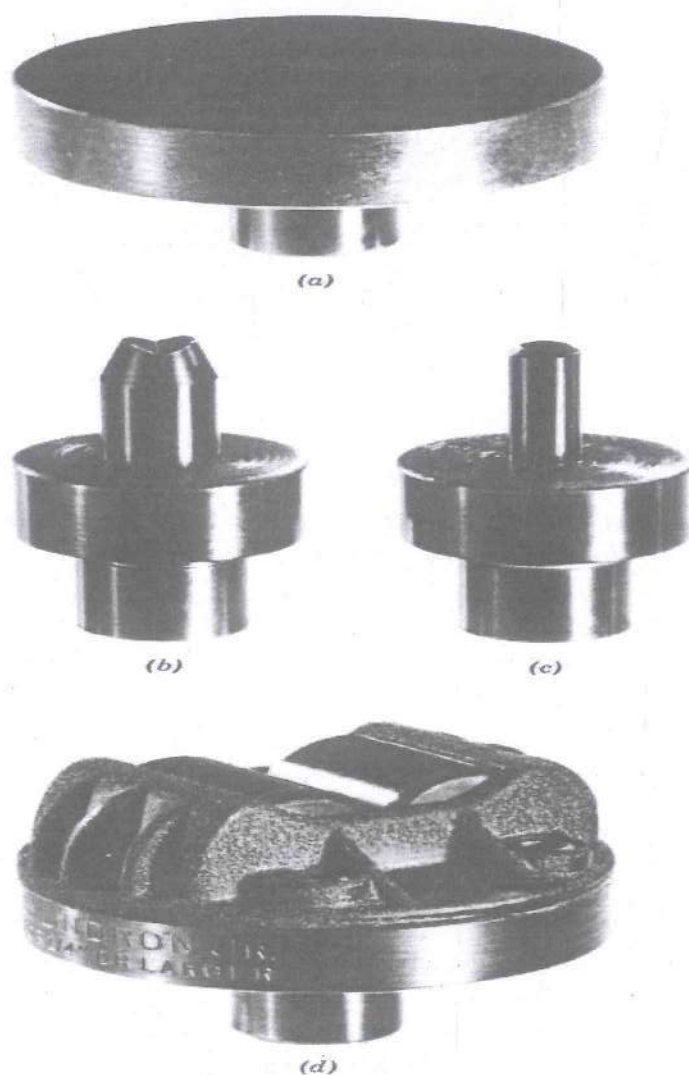


Figure 7. Basic anvils used with Rockwell hardness testers. (a) Plane, (b) shallow V, (c) spot, (d) culindron Jr. (Wilson Instrument Division of Acco)



Figure 8. Diamond spot anvil. (Wilson Instrument Division of Acco)

Surface preparation and proper use

When testing hardness, a surface condition is important for accuracy. A rough or ridged surface caused by coarse grinding will not produce results that are as reliable as a smoother surface. Any rough scale caused by hardening must be removed before testing. Likewise, if the workpiece has been decarburized by heat treatment, the remaining softer "skin" should be ground off the test area.

Error can also result from testing curved surfaces. This effect may be eliminated by grinding a small flat spot on the specimen. Cylindrical workpieces must always be supported in a V-type centering anvil, and the surface to be tested should not deviate from the horizontal by more than 5 degrees. Tubing is often so thin that it will deform when tested. It should be supported on the inside by a mandrel or gooseneck anvil to avoid this problem.

Several devices are made available for the Rockwell hardness tester to support overhanging or large specimens. One type, called a jack rest, is used for supporting long, heavy parts such as shafts. It consists of a separate elevating screw and anvil support similar to that on the tester. Without adequate support, overhanging work can damage the penetrator rod and cause inaccurate readings.

No test should be made near an edge of a specimen. Keep the penetrator at least 1/8 - in. away from the edge. A test block should be used every day to check the calibration of the tester if it is in constant use.

Useful Information

1. It is possible to convert from Rockwell to Brinell, and vice versa. For Rockwell C values (HRC) between -20 and 40, the Brinell hardness number is calculated by

$$BHN = 1.42 \times 10^6 / (100 - HRC)^2 \dots\dots\dots(4)$$

For HRC values greater than 40, use the following formula:

$$BHN = 2.5 \times 10^4 / (100 - HRC) \dots\dots\dots(5)$$

For HRA values that fall between 35 and 100, use the following:

$$BHN = 7.3 \times 10^3 / (130 - HRb) \dots\dots\dots(6)$$

2. It is worth mentioning that a statistical analysis of the measured values of hardness are necessary because more than four readings should be taken and different places should be considered for testing to see the variability of these readings. The statistical analysis comprises the calculation of the mean and standard deviation of the readings.

An average value is obtained by dividing the sum of all measured values of x by the number of measurements taken (n). In mathematical terms, the average $\bar{x}$ of some parameter xi is:

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n} \dots\dots\dots(7)$$

where n is the number of observations or measurements and xi, is the value of a discrete measurement.

Furthermore, the standard deviation s is determined using the following expression:

$$s = \left[\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n - 1} \right]^{1/2} \dots\dots\dots(8)$$

IV. The Experiment:

Material

- ◆ Steel and aluminum samples of different thickness and different treatments.

Equipment

- ◆ The Brinell testing machine.
- ◆ The Rockwell testing machine.
- ◆ Vickers or microhardness testing machine.

Procedure

The testing sequence of the Brinell hardness tester

- ◆ The desired load in kilograms is selected on the dial by adjusting the air regulator.
- ◆ The specimen is placed on the anvil. Make sure the specimen is clean and free from burrs. It should be smooth enough so that an accurate measurement can be taken of the impression.
- ◆ The specimen is raised to within $\frac{5}{8}$ in. of the Brinell ball by turning the handwheel.
- ◆ The load is then applied by pulling out the plunger control. Maintain the load for 30 seconds for nonferrous metals and 15 seconds for steel. Then release the load.
- ◆ Remove the specimen from the tester and measure the diameter of the impression.
- ◆ Determine the Brinell hardness number (BHN) by calculation or by using the table. Soft copper should have a BHN of about 40, soft steel from 150 to

200, and hardened tools from 500 to 600. Fully hardened high carbon steel would have a BHN of 750. A Brinell test ball of tungsten carbide should be used for materials above 600 BHN.

Brinell hardness testers work best for testing softer metals and medium-hard tool steels.

The testing sequence of Vickers hardness tester

- ◆ A specimen is placed on the anvil and raised by a screw until it is close to the point of the indenter.
- ◆ By tripping a starting lever, a 20:1-ratio loading beam is unlocked, and the load is slowly applied to the indenter and then released. A foot lever is used to reset the machine.
- ◆ After the anvil is lowered, a microscope is swung over the specimen, and the diagonal of the square indentation measured to 0.001 mm. The machine may also employ 1- and 2-mm ball indenters.
- ◆ Apply the load for 15 seconds
- ◆ Remove the specimen
- ◆ Calculate the hardness

The testing sequence of Rockwell hardness tester

- ◆ Using Table 2, select the proper weight and penetrator. Make sure the crank handle is pulled completely forward.
- ◆ Place the proper anvil on the elevating screw, taking care not to bump the penetrator with the anvil.
- ◆ Make sure that the specimen to be tested is free from dirt, scale, or heavy oil on the underside.

◆ Place the specimen to be tested on the anvil. Then by turning the handwheel, gently raise the specimen until it comes in contact with the penetrator.

◆ Continue turning the handwheel slowly until the small pointer on the dial gage is nearly vertical (near the dot). Now watch the long pointer on the gage and continue raising the work until the pointer is approximately vertical. It should not vary from the vertical position by more than five divisions on the dial.

◆ Set the dial to zero on the pointer by moving the bezel until the line marked "zero set" is in line with the pointer. You have now applied the minor load. This is the actual starting point for all conditions of testing.

◆ Apply the major load by tripping the crank handle clockwise.

◆ Wait 2 seconds after the pointer has stopped moving, then remove the major load by pulling the crank handle forward or counterclockwise.

◆ Read the hardness number in Rockwell units on the dial. The black numbers are for the A and C scales and the red numbers are for the B scale.

◆ The specimen should be tested in several places and an average of the test results taken, since many materials vary in hardness even on the same surface.

Student Notes

Hardness Testing

<i>Name</i>		<i>Section</i>	
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Results:

The hardness testing results

Al Specimen thickness (mm)	Brinell Hardness HB	Vicker's Hardness HV	Rockwell Hardness HR	Steel specimen Thickness	Brinell Hardness HB	Vicker's Hardness HV	Rockwell Hardness HR

Note: The test should be repeated four times for each thickness test.

Questions:

What is the effect of thickness on the results?

Find the limits of the hardness value using statistical tools described under the title "uncertainty" in this manual.

EXPERIMENT No. 9

Fatigue testing

I. Purpose

To introduce the student with the way of examining the behavior of the material under cyclic loading using a Wohler rotating fatigue apparatus.

II. Background

Review the sections in your materials science textbook covering the mechanical properties of materials.

III. Introduction

A component is often subjected to the repeated application of stress below the yield strength of the material. This cyclical stress may occur as a results of rotation, bending, or vibration. Even though the stress is below the yield strength, the material may fail after a large number of applications of stress. This mode of failure is called fatigue.

Failures caused by fatigue are found in many of the materials of industry. Some plastics and most metals are subject to fatigue in varying degrees. Fatigue is caused by a crack that is initiated by a notch, bend, or scratch that continues to grow gradually as a result of stress reversals on the part. The crack growth continues until the cross-sectional area of the part is reduced sufficiently to weaken the part to the point of failure. Even welding spatter on a sensitive surface such as a steel spring can initiate fatigue failure. Fatigue is greatly influenced by the kind of material, grain structure (anisotropy), and the kind of loading. Some metals are more sensitive to

sharp changes in section (notch sensitive) than others. Parts should be designed and fabricated to utilize directionality of grain flow. Fatigue is caused by a concentration of tensile stress that can often be corrected by a change in design (Figures 1a and 1b). Parts that are subject to stress reversals (cyclic stresses) can have their fatigue life extended considerably by raising compressive stresses on the surface (Figure 2). This can be done in several ways: by carburizing, nitriding, or surface hardening by induction or flame.

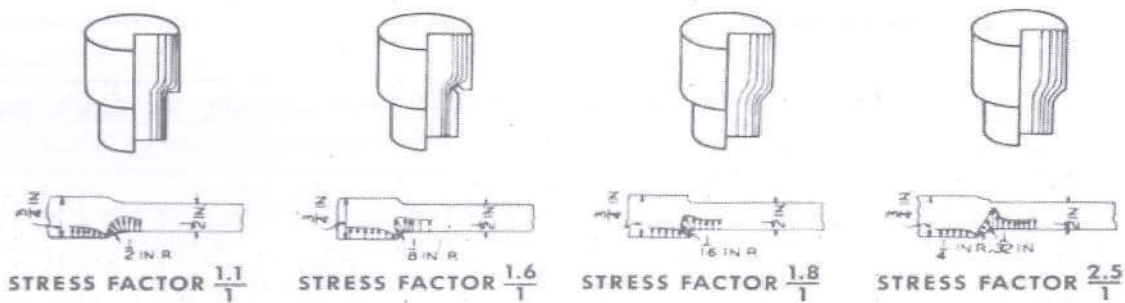


Figure 1a. Stress factors (Courtesy Steel Corporation).

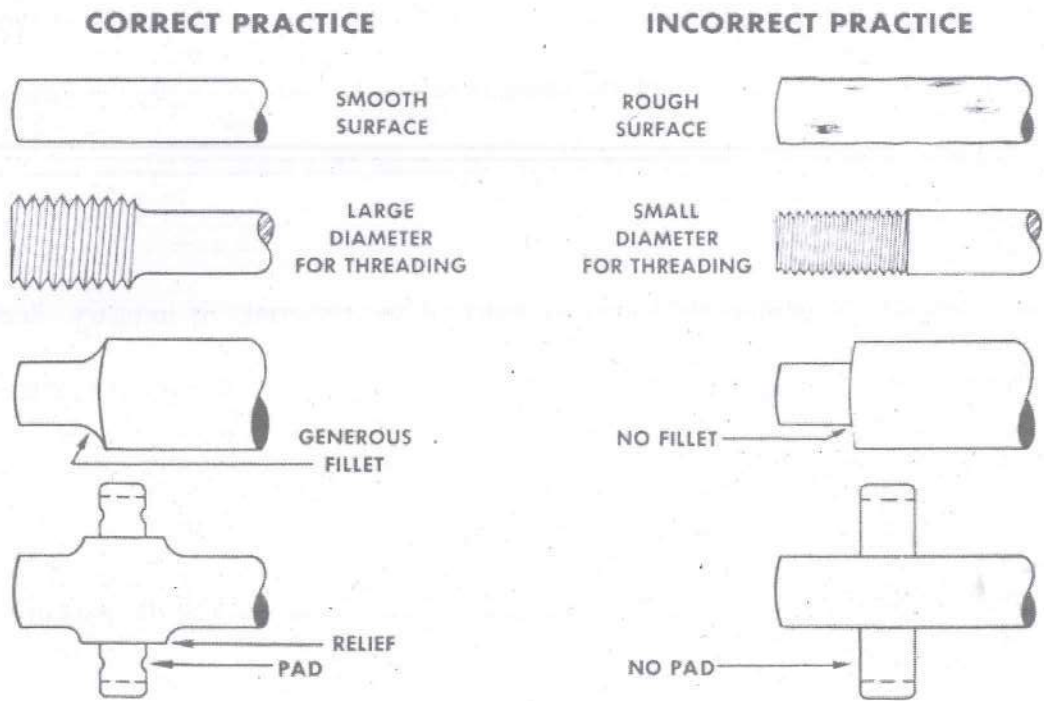


Figure 1b. Design consideration as stress is a function of design (Courtesy Steel Corporation).

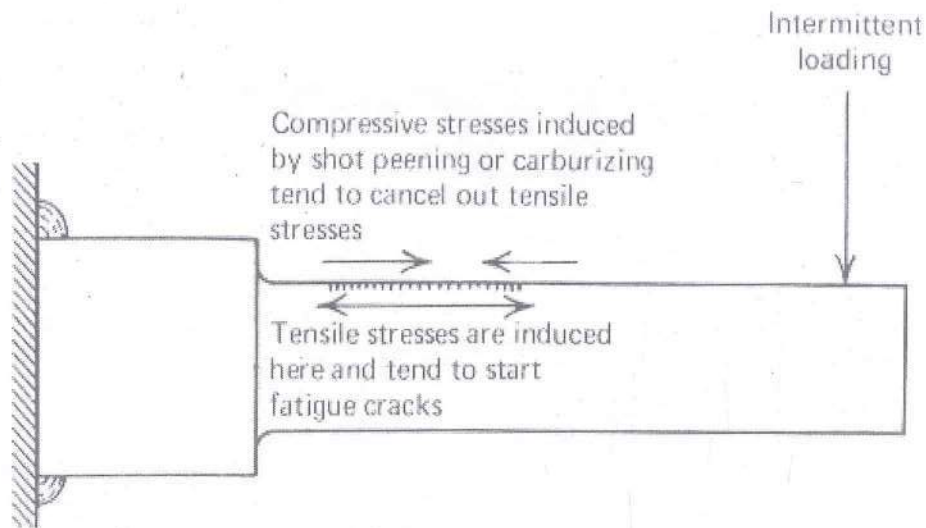


Figure 2. Fatigue failures are caused by tensile stresses on areas of stress concentrations. Compressive stresses induced by shot peening or carburizing tend to cancel out the tensile stresses and therefore reduce fatigue failure (Neely, 1989).

To increase fatigue life or the endurance limit, parts are sometimes subjected to shot peening to produce residual compressive stresses on the surface. A study of individual fatigue problems based on the service conditions and by direct observation of the failure can often lead to a conclusion that explains the cause or causes and suggests some corrective measures. The loading of the part can be high or low for its size; it can have high or low speed or stress reversals caused by misalignment. Vibration is often a cause of fatigue, the frequency and intensity being factors. Occasional overloads are also instrumental in initiating failure. Fatigue failures are usually characterized by three distinct surfaces or stages (Figure 3).



Figure 3. Classic example of fatigue in a shaft. The three distinct areas can be seen here.

Stage 1. A smooth surface with wave marks such as seen on clamshells. This area represents a slow progression of the initial crack.

Stage 2. A similar but rougher surface showing coarse wave marks progressing toward the center. The symbols and nomenclature related to fatigue failures are illustrated in Figure 4.

Stage 3. AcrySTALLine area showing the final sudden failure of the part. It is last portion that prompts the erroneous conjecture that the part has "crystallized".

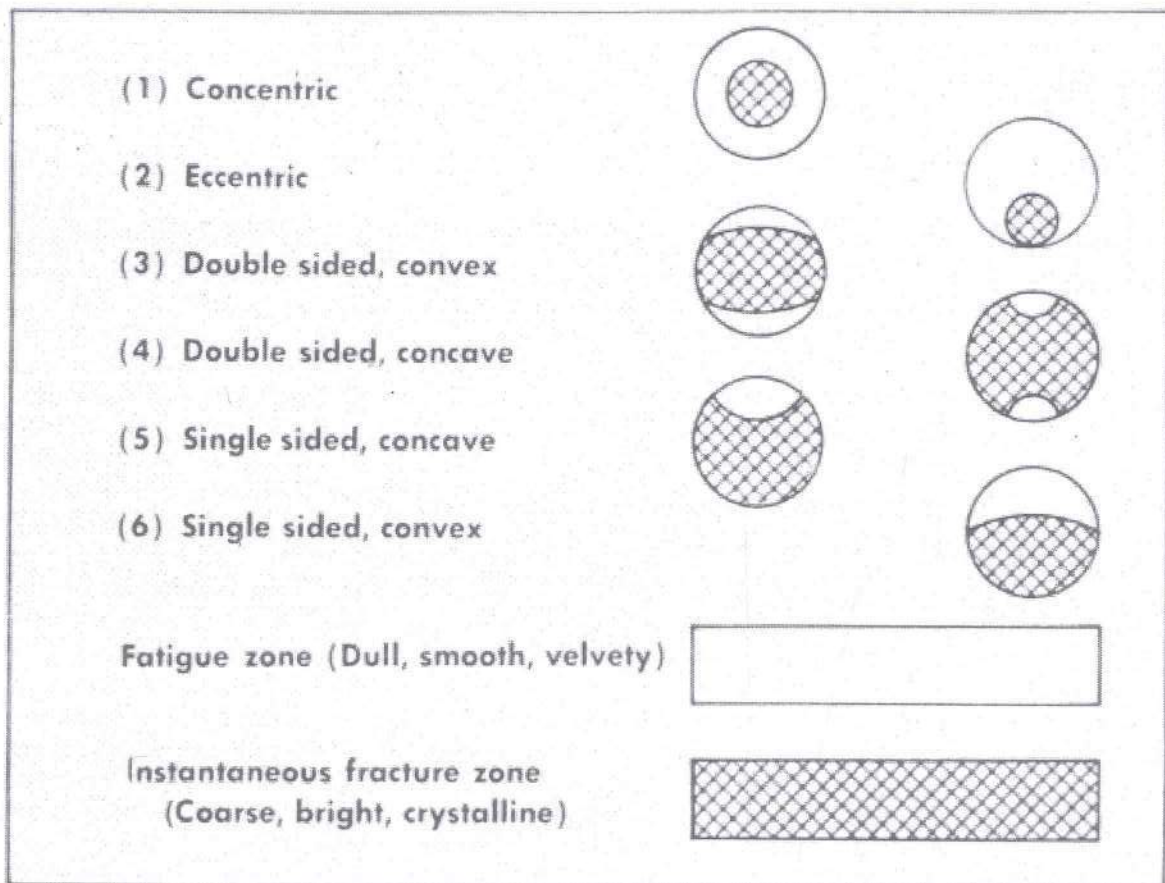


Figure 4. Symbols and nomenclature established by Bacon are useful for the designation of fracture appearance. (Courtesy Steel Corporation).

Stage 1 of the fatigue process may take months or years to progress to the more rapid Stage 2. The fatigue process in Stage 2 produces striations (as seen in the center portion), often on plateaus separated by shear bands or steps. Each striation is the result of a single stress cycle; however, a stress cycle does not always produce a striation. Stage 3 is often a brittle fracture, representing the final, sudden failure of the part.

Types of fatigue failures

Some of the more common fatigue fracture appearances associated with various bending conditions are illustrated in Figure 5. The following cases are examples.

Case \ Stress Condition	No Stress Concentration		Mild Stress Concentration		High Stress Concentration	
	Low Overstress a	High Overstress b	Low Overstress c	High Overstress d	Low Overstress e	High Overstress f
1 One-way bending load						
2 Two-way bending load						
3 Reversed bending and rotation load						

Figure 5. Fracture appearance of fatigue failures in bending. (Courtesy Steel Corporation).

Case I one-way bending load

No stress concentration

A small elliptically shaped fatigue crack usually starts at a surface flaw such as a scratch or tool mark. The crack tends to flatten out as it grows. It is caused by the stress at the base of the crack being lower because of the decrease in distance from the edge of the crack to the neutral axis. The degree of overstress in the part is indicated by the amounts of smooth-textured area as compared with the crystalline-textured area of the fracture. A large crystalline area indicates high overstress (Figure 5, 1-b). A smaller crystalline area indicates a lower overstress, which would require the greater number of cycles necessary to produce failure (Figure 5, 1-a).

Mild stress concentration.

If a distinct stress raiser such as a notch is present, the stress at the base of the crack would be high, causing the crack to progress rapidly near the surface, and the crack tends to flatten out sooner. The degrees of overstress by the relative areas of smooth and crystalline textures on the fracture surface are shown in Figure 5, 1-c and 1-d.

High stress concentration.

The smooth fracture growth can change from concave (as in 1-b) to convex as the rate of crack growth circumferentially at the surface exceeds the radial crack growth. In high overstress, the convex texture can occur extremely early in crack formation.

Case 2 two-way bending load

No stress concentration.

Cracks start almost simultaneously at opposite surfaces when the surfaces are equally stressed. The cracks proceed toward the center at similar rates and result in a fracture that is rather symmetrical (2-b of Figure 5). In low overstress conditions (2-a), cracks may not begin at the same time; consequently the fracture is less symmetrical.

Mild stress concentration.

Higher stress concentration and the increased rate of circumferential crack growth cause the fracture to flatten out more quickly. Rapid radial crack growth tends to promote a concave zone with a relatively small radius of curvature. As the relative rates of circumferential and radial crack growth tend to become equalized, the radius of curvature tends to increase.

High stress concentration.

In this. case, the circumferential crack rate increases rapidly, quickly exceeding the radial crack rate and the radius of curvature changes from concave to convex. The relative areas of smooth and crystalline textures discussed earlier also apply here.

Case 3 Reversed bending and rotational load

No stress concentration

As in Case 2, stress occurs at two extreme surface fibers. Usually the weaker area will fail first. The fracture tends to progress and flatten out from the initial small, concave crack. Eventually the fracture tends to become a straight line. The crack tends to propagate against the direction of rotation. With low overstress, the crack growth can proceed well beyond the center and promote circumferential growth prior to complete failure. With high overstress, the crack does not proceed as far as shown in 3-b of Figure 5.

Mild stress concentration

With high over-stress, the notch causes early crack formation and rapid crack growth around the periphery, and the crystalline zone is centrally located as shown in 3-d. Low over-stress tends to start the crack at the weakest point and the crystalline zone is moved away from the point of crack initiation (3-c). Extremely tough material will respond to these conditions with a fracture similar to 3-a.

High stress concentration

A combination of severe concentration and high overstress, such as a groove machined about the entire periphery with a sharp notch radius, causes cracks to start all around the circumference at the same time. The resultant crystalline failure is somewhat circular in appearance and centrally located (3-/). Lower overstress tends to

move the point of failure away from the central location (3-c of Figure 5). When a shaft is subjected to a torsional load, the maximum shear stress is equal to the maximum tensile stress. However, the corresponding two strengths in steel are not equal, the shear strength being approximately one-half the tensile strength. The shear stress, therefore, will reach the shear strength before the tensile stress will reach the tensile strength; therefore, a shear type of failure will result (Figure 6).

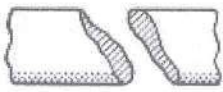
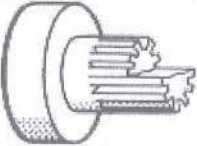
Kind of failure	Tensile	Transverse shear	Longitudinal shear
Pattern			
Variation	Star 	Offset 	Spline-type 

Figure 6. Basic types of torsional failure fractures.

One reason that transverse (crosswise) cracks are more prevalent than longitudinal cracks is that grinding or machining marks, which accentuate the probability of failure, are oriented in the transverse direction. The quality of surface finish is therefore very important. Splined shafts almost always produce a characteristic compound fracture. Fatigue cracks originate almost simultaneously at all the spline roots and grow until the shaft ruptures. The use of fibrous-type steels tends to increase this problem.

Basic concepts of the fatigue testing method

A common method to measure a material's resistance to fatigue is the rotating cantilever beam test. One end of a machined, cylindrical specimen is mounted in a motor-driven chuck. A weight is suspended from the opposite end (Figure 7).

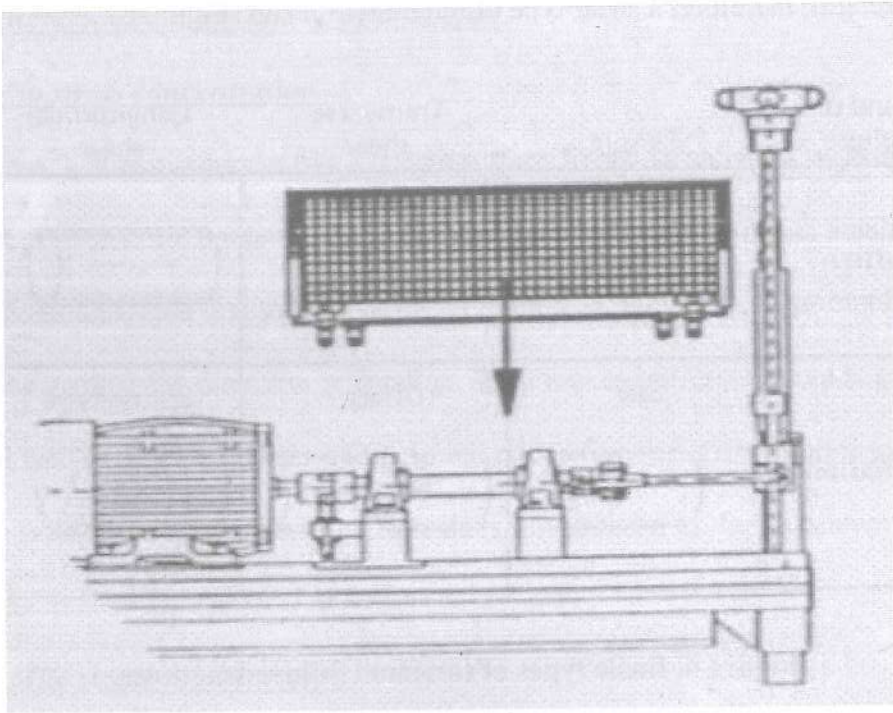


Figure 7. Schematic of fatigue testing machine.

The specimen (sample) is loaded as shown in Figure 8. The loading corresponds to a clamped bending bar under a concentrated force (F). This induces a triangular bending moment (M_b) in the sample. The bending moment is fixed but the specimen is rotating, it is loaded by an alternating, sign-shaped bending stress (Figure 9).

During rotation, the specimen initially has a tensile force acting on the top surface, while the bottom surface is compressed. After the specimen turns 90° , the locations that were originally in tension and compression have no stress acting on them. After a half revolution of 180° , the material that was originally in tension is now in

compression. Thus, the stress at any one point goes through a complete sinusoidal cycle from maximum tensile stress to maximum compressive stress.

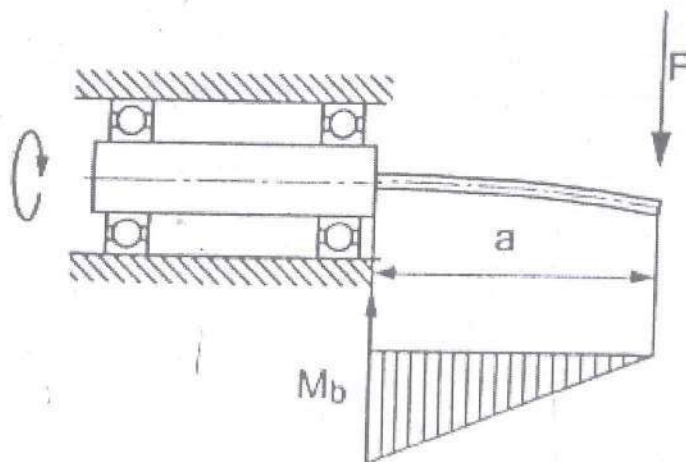


Figure 8. Schmatic loading of the sample

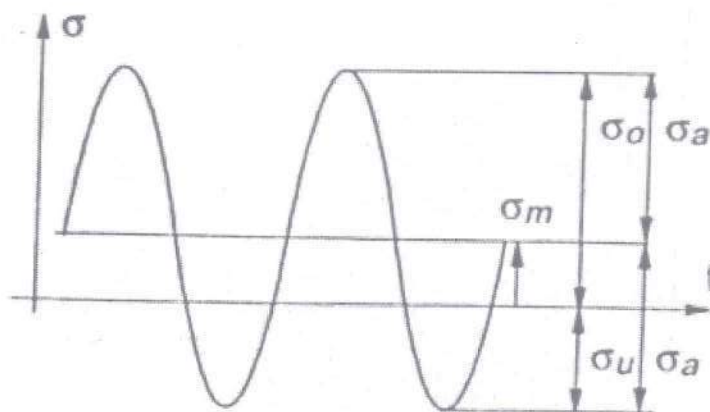


Figure 9. Alternating cyclic loading. σ_m -mean stress, σ_a -altrnating stress amplitude, σ_u -minimum stress, σ_0 -maximum stress.

Via various curvature radii and degrees of surface roughness of the specimen used, it is possible to examine the influence of the notch effect on fatigue strength. Based on this, three standard testing samples or bars of sizes as shown in Figure 10 are used for

fatigue testing. The stress (S) and the number of cycles (N) at failure are recorded and then plotted on an S-N work sheet with logarithmic scale (Figure 11).

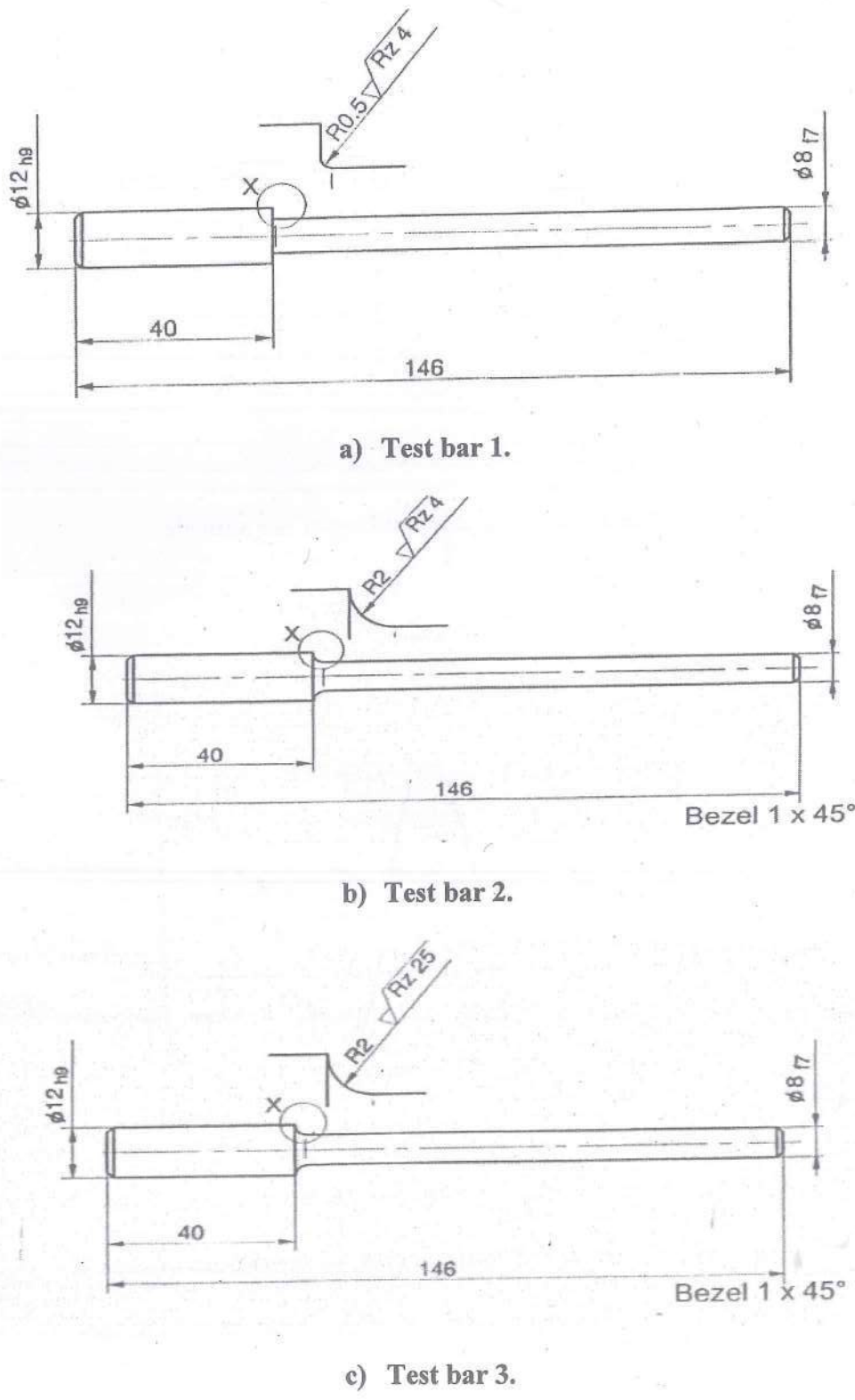


Figure 10. Standard testing bars

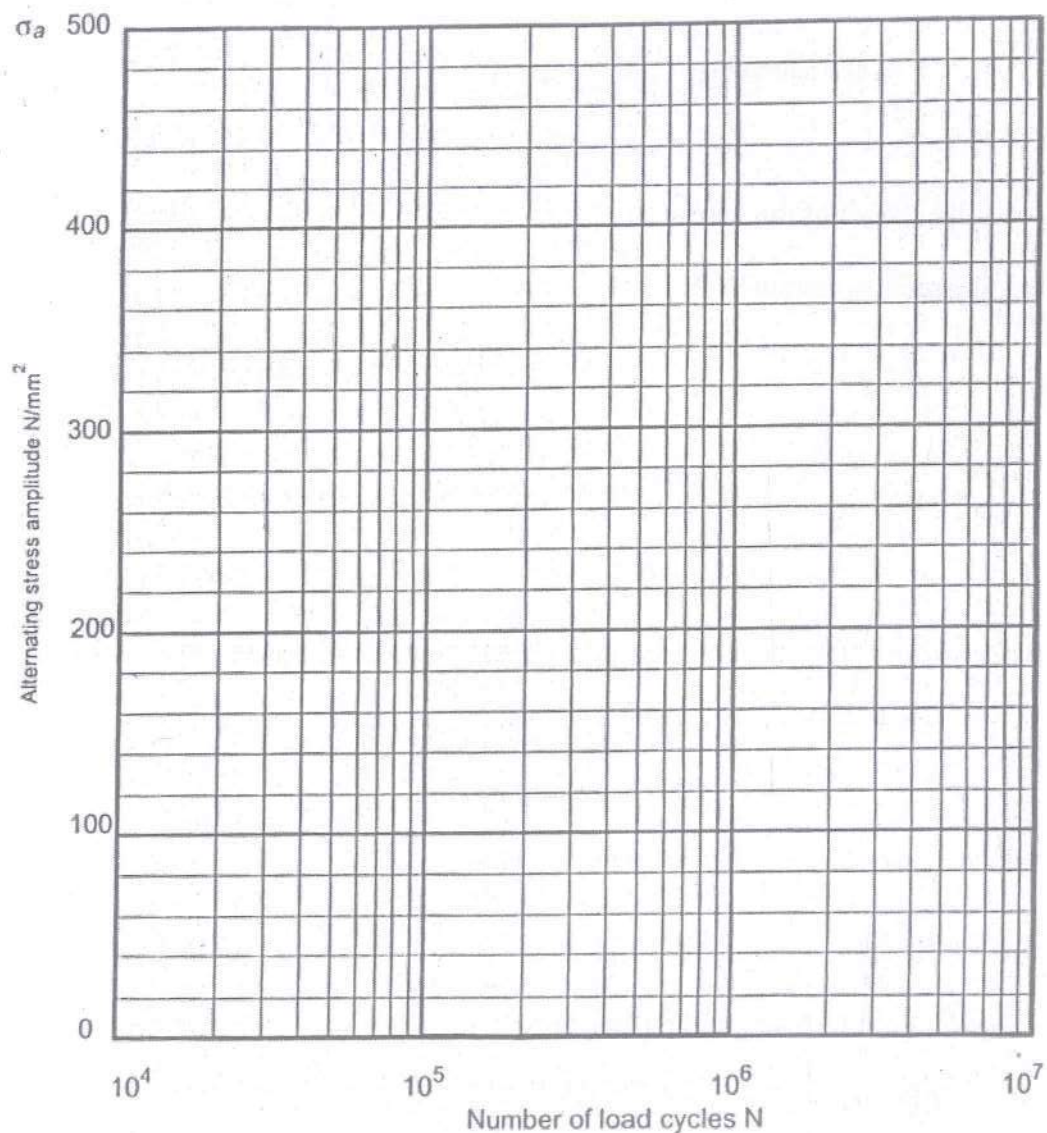


Figure 11. Stress – number of cycles at failure diagram (S-N curve).

The fatigue testing apparatus is working based on pure reversed bending stress without mean stress (mean stress = 0). For this reason it is only possible to determine fatigue strength under complete stress reversal (σ_w) with a revolving fatigue testing machine. It represents a special case of fatigue strength.

Referring to Figure 8, the bending moment is calculated as:

$$M_b = F.a \quad \dots\dots\dots(1)$$

Where:

M_b – bending moment

F – the load

A – the length of the lever arm

By using the section modulus (W_b):

$$\sigma_a = M_b / W_b \dots\dots\dots(2)$$

where:

σ_a - Alternating stress amplitude

$$W_b = \pi d^3 / 32 \dots\dots\dots(3)$$

The maximum bending stress acting on this type of specimen is given by:

$$\sigma_a = 32 F.a / \pi d^3 = 10.18 * F*a / d^3 \dots\dots\dots(4)$$

The endurance limit, fatigue life, and endurance ratio of the material can also be determined using the results of the test. These concepts can be found in your textbook and left for your own work.

IV. The Experiment

Material

A set of specimens of carbon steels.

A set of specimens of carburised carbon steel.

Equipment

WP 140 rotating fatigue testing apparatus

Procedure

♦ To carry out any meaningful fatigue experiment a consistent set of test specimens must be prepared. The typical standard specimen is shown in Figure 10. In this experiment a set of specimens of carbon steels and a set of specimens of carburised carbon steel were used. A minimum of 10 specimens for each Part is recommended. They must all be cut from one length of material. In addition at least one tensile test specimen must be taken if the lower yield or 0.2% proof stress, the ultimate strength, the elongation on five diameters across the fracture, and if possible the uniform elongation of the material being investigated are not known for the material.

♦ A set of bending stresses from say 0.9 to 0.5 of the yield or proof stress should be selected to match the number of test specimens for the complete experiment. The surface condition of the standard specimens should be noted (i.e. as machined, ground, polished).

♦ If the equipment is available, measure and record the surface roughness in the direction of the stress. Setting up is a reasonably simple operation provided it is done methodically. The objective is to align the specimen and loading arm with the axis of rotation to eliminate stresses due to eccentric whirling. Note

that one plain end of the necked specimen is slightly longer than the other; this end should be inserted into the chuck on the bearing block.

- ◆ Adjust the six grub screws until the specimen turns concentrically. Move the dial gauge to the other end of the specimen and check the concentricity..

- ◆ Now slide the loading arm onto the specimen making sure that the specimen is touching the bottom of the hole in the chuck. This fit ensures that the lever arm is the correct dimension.

- ◆ Select the bending stress for the test and apply the required weight on the load hanger. Set the revolution counter to zero, and fit the safety guard over the apparatus. With one finger ready to push the STOP button, use another finger to press the ON button. If the motor starts and all seems well note the time and make an estimate of when fracture might occur (allow one hour per 1/3million reversals).

- ◆ Should the motor not start adjust the micro switch downward slightly and try again.

- ◆ Normally the test terminates itself through the fracture of the specimen opening the micro switch and hence stopping the motor. As the onset of fracture approaches the specimen will bend more, and this may open the micro switch before complete fracture occurs. In this case move the micro switch down slightly and restart the motor.

- ◆ When the bending stress is at or below the endurance limit for mild steel it may be decided to stop the test at, say, 10 reversals (approximately 30 hours of running time). The result should be noted as NOT FAILED at the recorded count, and the test specimen must be labeled with its history and removed from the apparatus. The marks made by the twelve grub screws will show it is

no longer a virgin specimen. A similar situation may also arise when an aluminum alloy specimen is subjected to a low bending stress.

- ◆ Record the data

- ◆ Draw the S-N curve.

Part 1

Using a set of mild steel standard specimens, allocate the stress levels and tests throughout the class and proceed as above. Collate the results and plot them as they occur on a graph of stress range S against $\log_{10}$ number of reversals N .

Part 2

If time permits and the specimens are available, use a set of carburised specimens of the same material as the standard specimens, and proceed as in Part 1.

Results

Plot $\log S$ against $\log N$ on suitable graph paper and look for best-fit lines. If the results of previous classes are on record compare them with the current set of results. Apart from similarity between the graphs, a clever comparison can be made by using the ratio $R = \text{stress range } S / (\text{Ultimate strength of material instead of the actual stress range})$. Differences in surface finish will upset this.

Observations

The graphs for mild steel standard specimens should show an endurance limit. What proportion of the ultimate stress is it? How does it compare with typical elastic design stresses?

If the results contain repeated tests comment on their variability. In any event comment on apparent scatter of each whole set of results from the curves drawn

through them. If some results appear to be wild look at the fractured specimens to see if there are visible reasons.

Comment on the validity of any conclusions, which you add to the end of your report.

Student Notes

Fatigue Testing

<i>Name</i>		<i>Section</i>	
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Results:

Record the following data

Number of cycles for testing bar 1 under different loading

No.	Load (in N)	Stress σ_a (in N/mm ²)	Endurance N	Duration (min)

Number of cycles for testing bar 2 under different loading

No.	Load (in N)	Stress σ_a (in N/mm ²)	Endurance N	Duration (min)

Number of cycles for testing bar 3 under different loading

No.	Load (in N)	Stress σ_a (in N/mm²)	Endurance N	Duration (min)

Plot log S against log N and look for best-fit lines.

Questions:

Comment on the validity of any conclusions you included in your report.

EXPERIMENT No 10

Impact Test

I. Purpose

The student should recognize and understand the general procedures used to conducting an impact test, perform the necessary calculations related to impact testing, and recognize the expected impact test results.

II. Background

Review the sections in your materials science textbook covering the subject: Impact and Impact testing.

III. Introduction

Materials often exhibit different properties, depending on the rate at which a load is applied and the resulting strain that occurs. Most materials can withstand greater loads before failure if the load is applied gently over a longer period of time (static testing). If a smaller load is applied suddenly (dynamic testing), the material may fail and appear to have less strength. Because the properties of many materials are so strain-rate-dependent, tests have been standardized to determine the energy required to break materials under sudden blows. These tests are classified as impact tests. The general outcome of an impact test is the determination of the energy required to break the specimen.

Impact testing is used to measure the transfer of energy required to break a given volume of material. Impact strength is then an indication of how well a material can withstand shock loading. Because energy cannot be created or destroyed, the energy in the impact must be released through different channels. For example, energy can be consumed in the elastic deformation of the specimen, the plastic deformation of the specimen, friction between the moving parts, and other such actions. In the design of

structures and machines, an attempt is made to provide for the absorption of as much energy as possible through elastic action and rely secondly on some form of damping to dissipate it. In impact testing, the object is to use the energy of the blow to rupture the test specimen. Energy is defined as the ability to do work. Work is defined as a force operating through a distance. These two factors together give us the equation

$$W=FD.....(1)$$

where:

W: the work done (in ft-lb or N-m)

F: force applied (in lb or N)

D: distance through which the force was applied (in ft or m)

The object of the impact test is to determine the work done in breaking the object under a sudden blow. This can be accomplished by dropping a known weight on the material specimen from a known height. The equation involved in dropping a known weight from a measured height is

$$E = wh \text{ or } E = mgh \text{ (metric)}.....(2)$$

where:

E: the energy of the falling weight (in ft-lb or N-m)

w: weight of the object (in lb)

m: mass of the object (in kg)

h : the height of the object before falling (in ft or m)

g : 9.8 m/s²

The property of a material associated with rupture is called toughness. The causes of rupture are impact and force. Toughness depends chiefly on the ductility and strength of a material. Impact testing is an adequate measure of a material's toughness.

In making an impact test, the load can be applied in flexure, tension, compression, or torsion. Flexure loading is the most common, and tensile is less common. Compression and torsional loadings are used only in special circumstances. The impact blow can be delivered through dropping weights, swinging a pendulum, or rotating a flywheel. Some tests involve rupturing the test specimen in a single blow; others use repeated blows. In tests involving repeated blows, some are multiple blows of the same magnitude, whereas others, such as the increment-drop test, gradually increase the height from which the weight is dropped until rupture is induced.

Perhaps the most common tests are the Izod and Charpy impact tests. Both employ a swinging pendulum and are conducted on small, notched specimens broken in flexure. The two tests differ in the design of the test specimen and the velocity at which the pendulum strikes the specimen. In the Charpy test, the specimen is supported as a single beam, but in the Izod test, the specimen is supported as a cantilever. In these tests, a large part of the energy absorbed is taken up in a region immediately adjacent to the notch. A brittle type of fracture is often induced. Figure 1 shows the machine used to conduct the Izod and Charpy tests.

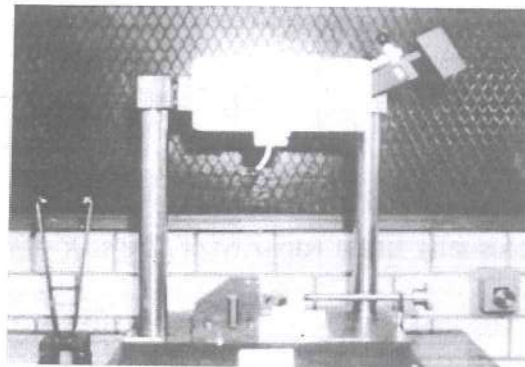


Figure 1. Testing machine for Izod and Charpy tests

In the Izod test, the specimen shown in Figure 2 is supported vertically, being held in a vise at the base. The standard test velocity for the Izod test is 11.5 ft/s. In the Charpy test, the specimen is mounted horizontally, being held at both ends as the pendulum

strikes the specimen. The standard test velocity for the Charpy test is 17.5 ft/s. The Charpy specimen is shown in Figure 3.

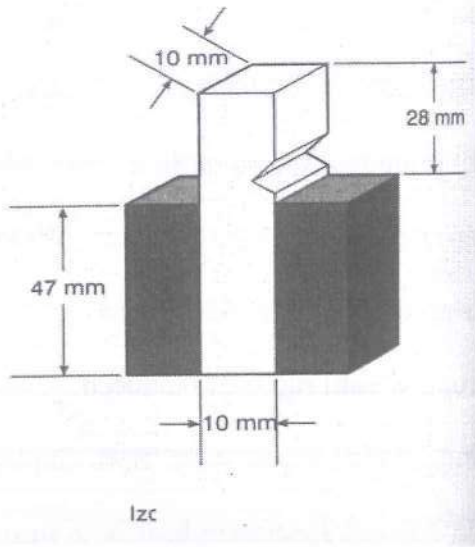


Figure 2. Izod test specimen

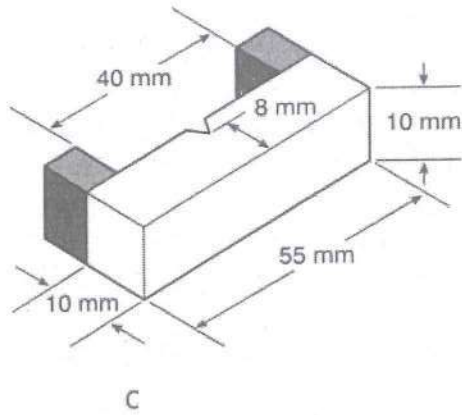


Figure 3. Charpy test specimen

The purpose of the notch in both specimens is to concentrate the stress, thus allowing the energy to be absorbed at a single point in the specimen. This facilitates the breaking of the specimen in a known region. Without the notch, the stress would tend to be evenly distributed throughout the specimen. The specimen would then plastically deform by bending rather than breaking. This would render the results invalid, because the purpose of the impact test is to determine the amount of energy required to break the specimen.

CharpyTest

A Charpy test machine usually has a capacity of 220 ft-lb for metals and 4 ft-lb for plastics (ASTM E 23). The pendulum consists of a relatively light, although rigid, rod or piece of channel, at the end of which is a heavy disk. This pendulum swings between two upright supports and has a rounded knife-blade edge at the end aligned so that it contacts the specimen over its full depth at the time of impact.

The standard test specimen is 10 x 10 x 55 mm, notched on one side in the center. Some tests require keyhole notches; others require U-shaped notches. The specimen is supported between two anvils so that the knife strikes opposite the notch at the midswing point. The pendulum is lifted to the initial release angle. It is then released and allowed to swing and strike the specimen.

The pendulum is set at a known angle, α , shown in Figure 4. Theoretically, if the pendulum does not encounter any resistance, it should swing to angle α on the opposite side. This theory discounts the effects of friction, which should be taken into account when calculating the energy required for rupture. When conducting the test, the pendulum is set to angle α . With the test specimen properly placed, the pendulum is released and allowed to swing freely. The pendulum gains momentum as it swings through its arc toward the specimen. When the pendulum strikes the specimen, breaking the specimen, it transfers some of its energy to the specimen. The pendulum then continues on to the opposite side of the machine at an angle β .

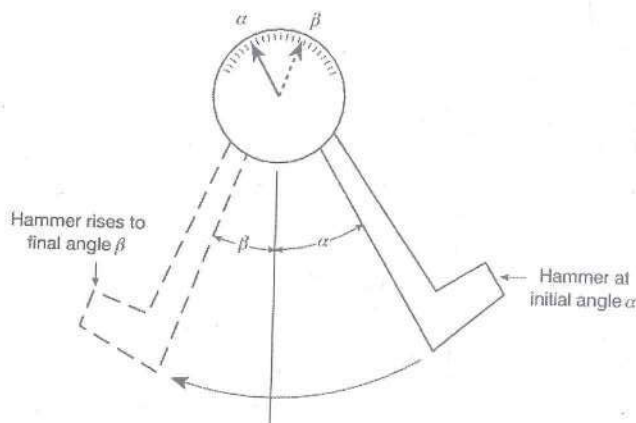


Figure 4. Impact test pendulum

Having recorded the weight of the pendulum, the length of the pendulum arm, and the initial and final angles (α and β), the following calculation can be made:

$$E = w(\cos \beta - \cos \alpha) \text{ or } E = mgr(\cos \beta - \cos \alpha) \text{ (metric)} \dots\dots\dots(4)$$

where:

E : weight of pendulum (in lb)

m : mass of pendulum (in kg)

g : 9.8 m/s²

r : length of pendulum (in ft or m)

α : initial angle (angle of fall)

β : final angle (angle of rise)

IzodTest

The common Izod test machine is made with a 120 ft-lb capacity. The test is similar to the Charpy test, although the specimen placement and features are different. In Izod impact testing, the pendulum strikes the front face of the test specimen, just the reverse of the Charpy impact test. In the Izod test, the specimen is 10 x 10 x 75 mm, having a 45° notch cut 2 mm deep. The impact strength of the specimen is based on the angle of rise after rupture occurs. The energy in foot-pounds can be read directly from the dial on the tester.

IV. The Experiment

Material

Three types of materials are used in this test: Killed low carbon steel, Three percent nickel low carbon steel, and Six percent nickel low carbon steel.

Apparatus

The creep machine is shown in Figure 1.

Procedure

- Prepare the items that are required in impact testing, which are the foundation, anvil, specimen supports, specimen, striking mass, and velocity of the striking mass.
- The specimen should be supported firmly and in the correct position throughout the test.
- Determine the weight and the length of the arm of the pendulum
- Determine initial angle (α) of the pendulum
- Determine final angle (β) of the pendulum
- The pendulum should be supported to reduce the lateral play, or restrain, and friction that may be felt as it swings in an arc toward the test specimen.
- The release mechanism should be constructed to reduce any binding, acceleration, or vibratory effects.
- The kinetic energy is determined from, and controlled by, the mass of the pendulum and the height of free fall measured from the center of the mass.

Student Notes

Impact test

<i>Name</i>		<i>Section</i>	
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Results:

Calculation and comparison of results.

Non destructive Testing

I. Purpose

To introduce the student to some non-destructive testing methods and to investigate the availability and occurrences of defects within the given specimen.

II. Background

Review the sections in your materials science textbook covering Failure-origin, detection, and prevention.

III. Introduction:

All metallic materials are imperfect, but this is only of concern if imperfections adversely affect intermediate processing or the use of the finished product. In order to detect imperfections, some form of testing is necessary. The objective of all testing is to insure that materials and components perform in the way expected. If the testing does not destroy the material in any way it is known as non-destructive testing (NDT).

Destructive testing has the following limitations:

- 1- A certain amount of the material has to be allocated for the testing, some of which, however, may be subsequently usable as scrap.
- 2- Special test-pieces and specimens may have to be prepared.
- 3- Components may be damaged and have to be scrapped. The more work which has been carried on the component before testing, the greater the total cost involved in the testing.
- 4- In order that the test results procedures be standardised, test-pieces and specimens are taken from products must be such that these are representative of the bulk material. Test-pieces must be then carefully prepared according to standard methods to ensure reliable and meaningful results.

These limitations and the development of highly stressed components such as aluminum alloy castings and forgings in aircrafts, and welded vessels in high-pressure steam and nuclear energy plant, focused attention on means of detecting flows in the finished article without destroying or damaging any part of it. Such non-destructive tests include the following techniques: Visual, penetrant, magnetic particle, electrical, acoustic and radiological methods.

Non-destructive testing may be broadly classified as follows:

Detection of surface flows:

- I. Visual inspection.
- II. Magnetic particle methods.
- III. Electrical methods.

Detection of internal flows:

- I. Acoustic methods: Sonic and Ultrasonic methods.
- II. Radiological methods which may be subdivided into: x-ray examination and gamma-ray examination.

Visual inspection

1. Fluorescent-penetrant inspection

Invisible surface cracks, porosity, and other surface defects are also found by this method in metals such as iron, steel, aluminum, bronze, tungsten carbide, and nonmetals such as glass, plastics, and ceramics. Fluorescent-penetrant inspection is widely used for testing and inspection on these materials. Zyglo, the Magnaflux Corporation copyrighted name for this method, is similar to Magnaglo in the use of black light to make the defects glow with fluorescence (Figure 1). Once applied, Zyglo penetrant is drawn into every defect no matter how fine or deep, but it must be given the time to do so. The length of time depends on the type of defect. The surface

film is rinsed off with water, developer is applied to draw out the penetrant, and the part is inspected for defects under black light where cracks and other flaws will fluoresce brilliantly. Zyglo systems are available in many sizes, ranging from handportable test kits to huge automated systems.

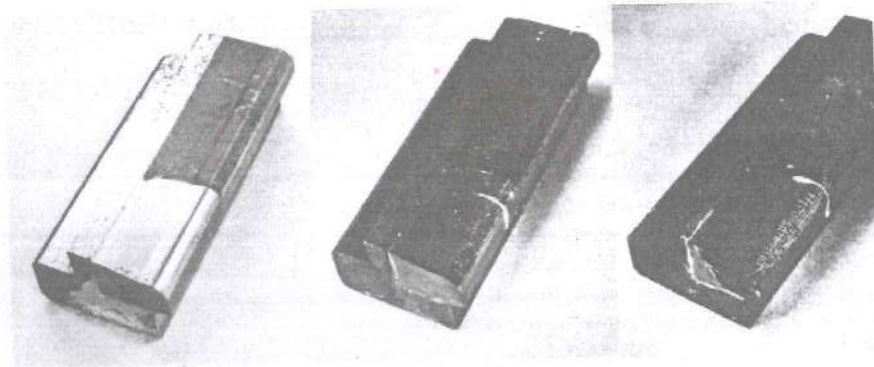


Figure 1. Freshly reground carbide tipped tools as they appear; (left) – normal visual inspection, perfectly good; (center)- test results with Zyglo penetrant; and (right)- with supersensitive Zyglo pentrex. All cracks however small are found.

(Courtesy of Magnaflux Corporation)

2. Dye penetrant

A similar method of nondestructive testing using dye penetrants is visual inspection, but without the black light and fluorescent penetrant. As with Zyglo, it may be used on almost any dense material. This method, called Spotcheck by the Magnaflux Corporation, works as follows:

1. Surfaces must be clean and dry prior to soaking.
2. Dye penetrant is applied to the defect and allowed to soak. Soaking time should be sufficient to get the penetrant into fine cracks.
3. Remove excess dye penetrant, but do not rinse out cracks.
4. The developer is applied. Allow enough time for the developer to find very small cracks.

5. Inspection shows a bright colored indication marking the defect.

Spotcheck (Figure 2) is available in sealed pressure spray cans or for brush or spray gun application. Its advantages are portability for remote uses (Figure 3) or for rapid inspection of small sections in the shop, low cost, and ease of application.



Figure 2. Spotcheck is used to locate fatigue crack in punch press frame.

(Courtesy of Magnaflux Corporation)

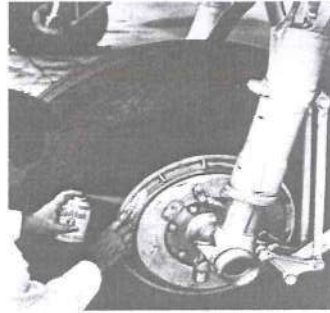


Figure 3. Spotcheck is used to find dangerous crack in an aircraft wheel.

(Courtesy of Magnaflux Corporation)

Magnetic particle method (electro magnetic Yoke)

The magnetic particle method of inspections is a procedure used to determine the presence of defects at or near the surface of ferromagnetic objects. It is based on the principle that if an object is magnetized, irregularities in the material, such as cracks or non-metallic inclusions, which are at an angle to the magnetic lines of force, cause an abrupt change in the path of a magnetic flux flow through the piece normal to the irregularity, resulting in a local flux leakage field and interference with the magnetic lines of force (Figure 4). This interference is detected by the application of a fine powder of magnetic material, which tends to pile up and bridge over such discontinuities. Under favorable conditions, surface crack is indicated by a line of fine particles following the outline of the crack, and a subsurface defect by a fuzzy collection of the fine particles on the surface near the defect.

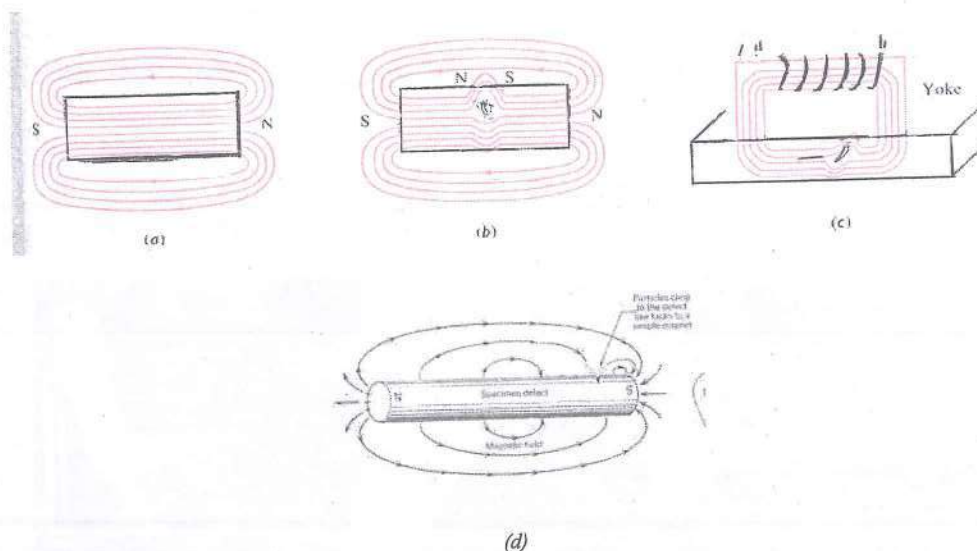


Figure 4. (a)- Normal lines of magnetic flux in a defect-free material; (b)- A flaw in a ferromagnetic material that causes a disruption of the normal lines of magnetic flux; (c)- Effect of flaw orientation on disruption of lines of flux; (d)- Surface cracks made visible by magnetic particles

High frequency eddy current method

Eddy currents are alternating currents which are electro-magnetically induced in electrical conductors, and which flow in closed loops. To generate eddy-currents a coil, energized by an alternating current supply, is placed close to the metal under test, this induces in the metal, alternating currents mirroring the current flow in the coil. These eddy currents can be detected by reverse process.

In eddy-current testing, a search coil carrying (AC) is moved over the surface of the component, under test and any change in the electrical properties of the coil is assumed to indicate the presence of flaws. The method is only suitable for the detection of surface and near surface defects in products of uniform section such as bars, rods, tubes and wire. A schematic representation of the through coil and probe methods for eddy current inspections is shown in figure 5.

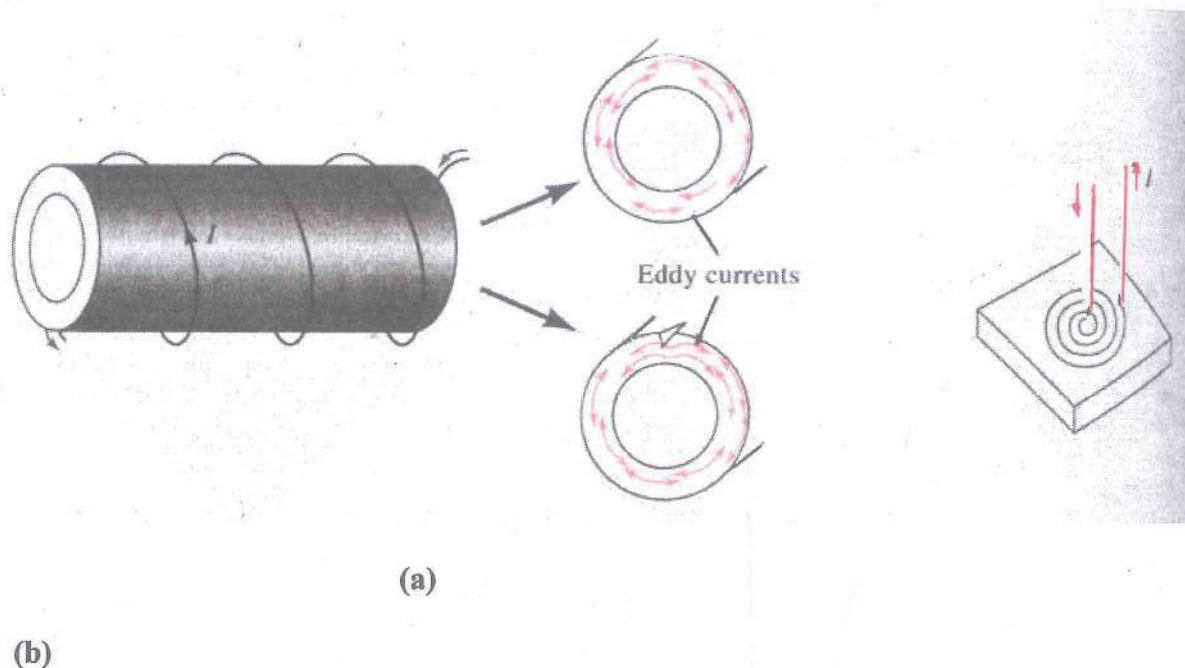


Figure 5. A schematic representation of the (a)- through coil and (b)-probe methods for eddy current inspections.

In the non-ferrous wrought metal industries, eddy current testing is the most widely used non-destructive testing technique. It is also occasionally used in steel industries, but the ferro-magnetic nature of steel complicates the method. An advantage of eddy-testing is its speed. Rods and tubes at throughput speeds up to 3.5 m/s and fine wire up to 50m/s. No coupling medium is needed.

Ultrasonic testing

Sound is produced when an object vibrates and can be propagated only through a medium; it cannot travel through a vacuum. Sound is described in terms of its frequency, i.e. the number of complete vibrations (cycles) which occur in one second. The unit of frequency is the hertz (Hz) so that a frequency of 1Hz means 1 cycle/sec. The full range of sound frequencies includes both a sonic range between 16-20 kHz (which is audible to human ear) and an ultrasonic range of 20 up to 1000 MHz or more. Ultrasonic testing uses frequencies in the range of 0.5 to 20 MHz. Ultrasonic vibrations, in contrast to audible vibrations, are not easily transmitted through gases. However, ultrasonic vibrations will

readily pass through liquids and solids and can travel very long distances provided the media are homogeneous. Discontinuities and interfaces (particularly with air) cause ultrasonic vibrations to be almost completely reflected.

Flaw detection

The ultrasonic beam is introduced to the product under test by direct contact between the crystal probe and the product. A film of coupling medium(usually oil) must be interposed between the probe and the product because even a very thin gap(less than 10^{-3} mm) would cause total reflection. It is not necessary to machine the surface of the product, but scale, rust or any other foreign matter which would interfere with the close contact of the probe with the surface should be removed. Ultrasonic waves may be used in two ways for flaw detection:

- i) Pulse-reflection method.
- ii) Transmission method.

The pulse-reflection method

Is by far the most commonly used. In this technique a pulse of ultra sonic waves is introduced to into the component under the test and the time which elapses between the transmission signal and the return of the reflected echo from the back wall of the component, or from some intermediate defect, is measured. For a particular material this time is proportional to the path length of the waves, so that time is related to the distance (Figure 6).

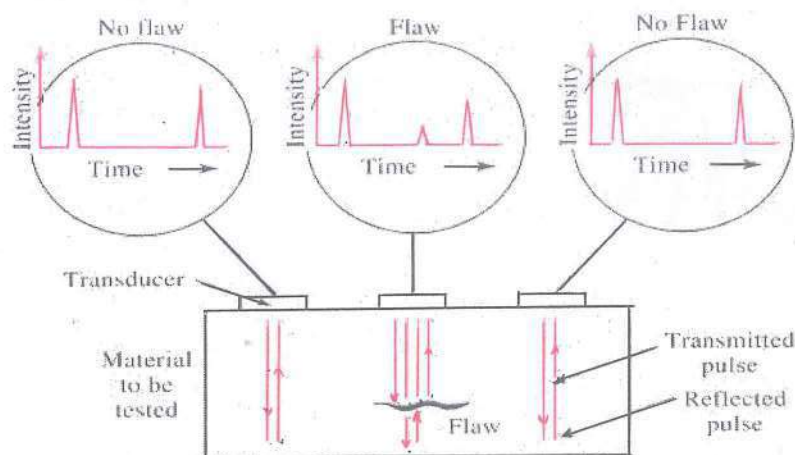


Figure 6. The pulse-echo ultrasonic test

This method has the advantages that the distance of the defect from the transmitting-receiving crystal (transceiver) can be accurately measured and also that this measurement can be made for just one side of the component.

The indication of the transmitted pulses and reflected echoes, and the measurement of the time interval between them, is carried out using a cathode-ray oscilloscope. The apparatus can be calibrated directly in units of the length, and the distance from the surface in contact with the probe to the flaw can be read in millimeters.

Transmission method

A continuous ultrasonic beam is used. Two probes, one a transmitter and the other a receiver, are positioned on opposite sides of the component under test and the ultrasonic beam passes from one to the other as shown in (Figure 7). Two peaks only appear on the cathode-ray oscilloscope, the one on the left is the transmission peak and the right hand one showing the intensity of the beam reaching the receiver. In this case of a perfectly sound component, the two peaks are of equal height, but, when a defect is present, the peak on the right is reduced in height, because some of the energy of the beam is reflected from the discontinuity to the transmitter.

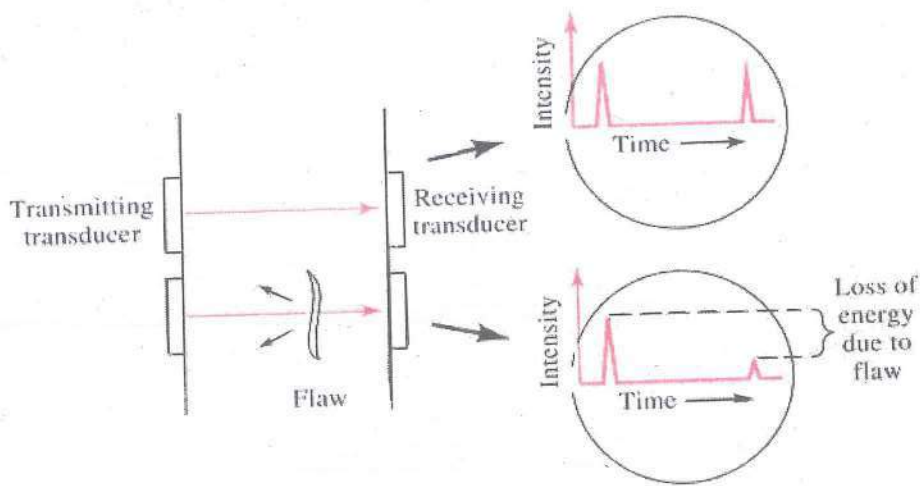


Figure 7. Through transmission ultrasonic test

In the transmission method the two surfaces of the component on which the probes are used must be parallel, because if the ultrasonic beam reaches the opposing face at an angle, there will be refraction in addition to reflection.

The pulse-reflection is preferred for the following reasons:

- i) A single transceiver only is required.
- ii) Only one surface of the component under test is used.
- iii) The backwall of the component need not to be parallel to the opposite face

Radiographic testing

Radiographic testing utilizes the ability of X rays (Figures 8) or gamma rays (Figures 9) that are emitted from radioactive materials, such as radio active radium or cobalt 60, to pass through solids. The test results are determined from a radiograph, which is a film exposed to radiation that has gone through the test materials.

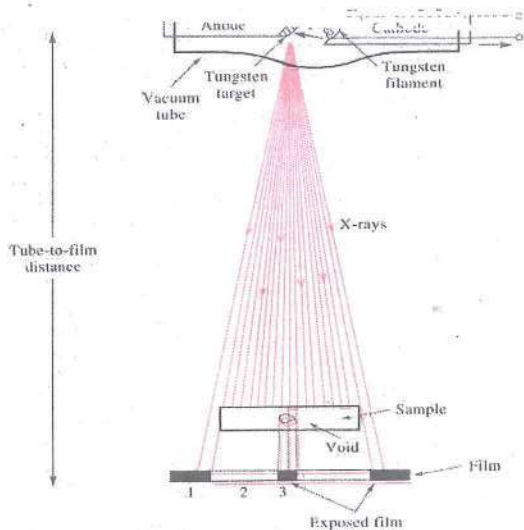
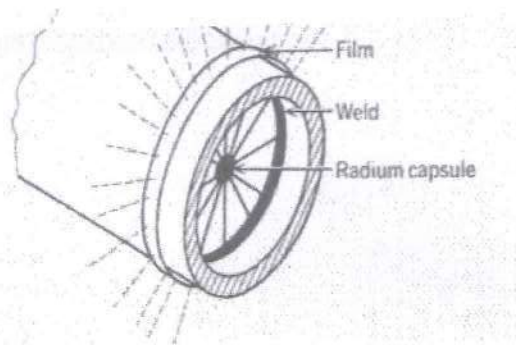


Figure 8. How X-ray inspection works



**Figure 9. How gamma-ray inspection works
(Machine tools and Machining Practice)**

Shadows on the radiograph reveal defects, since the radiation will pass through a void, crack, or area of lower density at a greater intensity and will appear darker on the negative (Figures 12a and 12b). This testing method is widely used for forgings, castings, welded vessels including welds on pipelines, and corrosion analysis on pipelines and structures. Portable equipment is used for field inspection. Since there is a radiation hazard, only trained technicians should use the equipment. The test is quite sensitive and provides a permanent record. However, a new, low-intensity, portable X-ray device has been developed that uses low-level radioactive isotopes. A person can operate this device for several years and incur no more radiation exposure than an annual X ray, according to the manufacturers. Objects can be viewed on a screen in real time (instantly) and can also be recorded on a Polaroid film. Portable X-ray analyzers are also used for verifying and sorting metals for alloy content and impurities. These devices are ideal for sorting scrap metals for producing high-integrity steel and nonferrous metals. A radiation detector may be used to determine material thickness, since the radiation that passes through the test material decreases

as the thickness increases. A moving, continuous strip of material can then be constantly monitored for thickness without any physical contact with the radiation source or detector.



Figure 12a. The gear looks perfectly sound on the outside surface.(Neely, 1989)

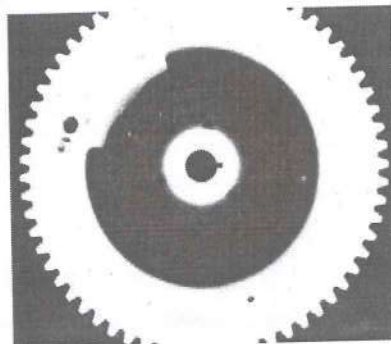


Figure 12b. A radiograph of the gear in Fig.12a. It reveals considerable porosity (black spots near the hub are holes in the metal).(Neely, 1989)

IV. The Experiment

Magnetic particle method (electro magnetic Yoke)

Material

- ◆ steel tube.
- ◆ Black magnetic ink
- ◆ Iron magnetic powder

Equipment

- ◆ Electro-magnetic yoke.

Procedure

- ◆ Clean inspected area of specimen with steel brush and wipe off with cloth.
- ◆ Place yoke on test piece perpendicular to direction of suspected cracks.
- ◆ Energize yoke magnetic field will form in test piece.
- ◆ Apply magnetic ink or powder while yoke is still energized.
- ◆ Indications will form immediately.

High frequency eddy current method

Materials

- ◆ Aluminum and steel specimen

Equipment

- ◆ Locator UH high frequency eddy current unit probes.

Procedure:

Setting up

- ◆ Connect probe required and set the frequency switch to match.
- ◆ Set type of alarm condition required
- ◆ Set the metal selector-switch to match the metal to be tested.

- ◆ Set alarm level required.
- ◆ Switch to "ON".
- ◆ Place probe on surface of sound part of the metal.
- ◆ Press and release "TRAIN" bottom. Lower L.E.D lights up. Raise probe until upper L.E.D lights. Lower probe to surface, raise probe again until upper L.E.D lights, lower probe to surface. "TRAIN" period is ended when both L.E.D 's are extinguished.
- ◆ Movement upwards during "TRAIN" should be only a fraction of a (mm). Downward movement to the surface of the metal should be gentle.
- ◆ Probes with flat ends should be held accurately perpendicular to the metal surface during the train procedure.
- ◆ Ideally the probe should be raised and lowered about twice/sec during the train period but this is not critical. After two or three cycles, the probe may be left resting on the metal until the train period is over.
- ◆ Ensure that lift-off is compensated by raising the probe slightly by inserting a plastic spacer (0.1-0.2mm) and check that meter deflection is less than 20% when sensitivity is set at 5.

Note that GMH2 flat-end shielded probes will compensate only over very short distances, so lift-off is best checked by rocking the probe by say +/- 15 degrees.

- ◆ The unit is now compensated for lift-off and will indicate surface breaking cracks. Sensitivity may be set by training on the calibration block of similar metal and setting the deflection for a given slot depth e.g 80% deflection for 0.5 mm slot.

Operation

- ◆ Move the probe lightly across the work piece surface , maintaining the probe approximately perpendicular to the surface of the sample.
- ◆ A crack will be indicated by a sharp kick on the meter.
- ◆ The meter deflection is at a maximum when the probe head is over the center of the crack. The magnitude of the deflection indicates the severity of the crack.

Required

- ◆ Draw a schematic diagram of the apparatus used.
- ◆ Make a sketch of the used specimen.
- ◆ Show the obtained results for the specimen used.

Ultrasonic testing

Equipment

- ◆ Ten-eleven SG ultrasonic unit and transducer.
- ◆ Ferrous and non-ferrous specimens.

Procedure

Setting-up

- ◆ Connect transducer as a Tx “transmitter” or Rx “receiver”.
- ◆ Set gain required.
- ◆ Set range required.
- ◆ Turn on the unit.
- ◆ Set focus needed.
- ◆ Place lubricant “oil, water” on the surface of the workpiece.
- ◆ Place transducer on surface of the metal.

Operation

- ◆ Move the transducer gently across the workpiece surface.
- ◆ A discontinuity “crack, hole” will be indicated by a pip on the oscilloscope.
- ◆ The distance between the blips (pips) is proportional to the depth of the defects.

Required

- ◆ Draw a schematic diagram of the apparatus used.
- ◆ Make a sketch of the used specimen.
- ◆ Show the obtained results for the specimen used.

Write your results and observations in a report form.

Student Notes

Non-destructive Testing

<i>Name</i>		<i>Section</i>	
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Results:

Magnetic particle method:

Sketch of the used specimen and the obtained results for it

High frequency eddy current method.

Sketch of the used specimen and the obtained results for it

Transmission method

Sketch of the used specimen and the obtained results for it.

Section IV. Solid solution strengthening

EXPERIMENT No.12

Construction of Equilibrium Phase Diagram From Cooling Curves

I. Purpose:

To introduce the student with the way of constructing phase equilibrium diagrams from the cooling curves.

II. Background:

Review the section in your materials science textbook covering the subject: solid solution strengthening and equilibrium phase diagrams.

III. Introduction:

Equilibrium phase diagrams are graphical representations that can be used to indicate the phases of the alloy that present at any selected temperature, the composition of each phase, and the amount of each phase for the desired temperature and composition. Equilibrium phase diagrams have wide applications in melting operations, casting, welding and heat treatment processes.

Many techniques are used for the experimental determination of phase diagrams. The widely used technique is the thermal analysis. In this method cooling curves of different proportions of elements (0 – 100%) are obtained where the rest points obtained when the temperature to time ratio changes indicate phase and lattice changes (microscopically). Studying a particular cooling curve yields data related to a particular combination of two or more metals for the entire temperature range through which the alloy cools. And the cooling curves of all possible combinations and temperature constitute the equilibrium phase diagram.

This method is based on the idea that if a hot system is allowed to cool freely in a constant environment, any marked change in its rate of cooling at some temperature indicates an evolution of heat due to a change in the system itself. To determine the cooling curve, a thermally-logged crucible of the alloy (often in a vacuum or an atmosphere of inert gas) is allowed to cool slowly. Its temperature is measured at frequent and regular intervals of time by means of a sensitive thermocouple (platinum, rhodium, chromel-alumel, copper-constantan or silver-constantan). The hot junction is protected by a thin refractory sheath and immersed in the metal, while its cold junctions immersed in melting ice and connected to a potentiometer. In this way cooling curves can be determined.

The cooling curve is affected by composition. If a pure metal is heated and then cooled very slowly, its cooling curve may be plotted as shown in Figure 1(a). The curve *ab* represents the cooling of the melt. At temperature *T* and at point *b*, the pure metal starts to the solid pure metal. Note that curve *bc* is level and that the phase change occurs at one temperature *T*. From *c* to *d* the solid metal undergoes cooling.

In Figure 1 (b) two metals have been heated to point *a* where both are liquid and are dissolved in each other. On slow cooling they remain liquid until they reach *T*, at which temperature solidification starts. At *t*₂ the solidification has been completed, and the entire mixture is a solid solution. The cooling of the solid continues till point *d*. Point *b* is the point where crystallization begins and is referred to as the liquidus point. Point *c* is the point of the curve where final solidification takes place. It is referred to as the solidus point. Another type of cooling curve is one that exhibits characteristics as shown in Figure 3(c). This occurs when two metals *A* and *B* are soluble in the liquid state but insoluble in the solid state. In this mechanism the liquid solution starts to cool at *a*. At *b*, pure metal *A* starts to form and precipitate out of the

liquid phase. As the temperature drops from T_1 to T_2 , pure metal A continues to precipitate. As metal A precipitates out, the ratio of B to A increases and we say that the liquid remaining becomes richer in B . This mechanism of freezing results in the slope of the curve bc .

At c the freezing is that of the remaining metals of A and B . The temperature remains constant until all solidification is complete at d . Line de represent the normal cooling of the solid phases.

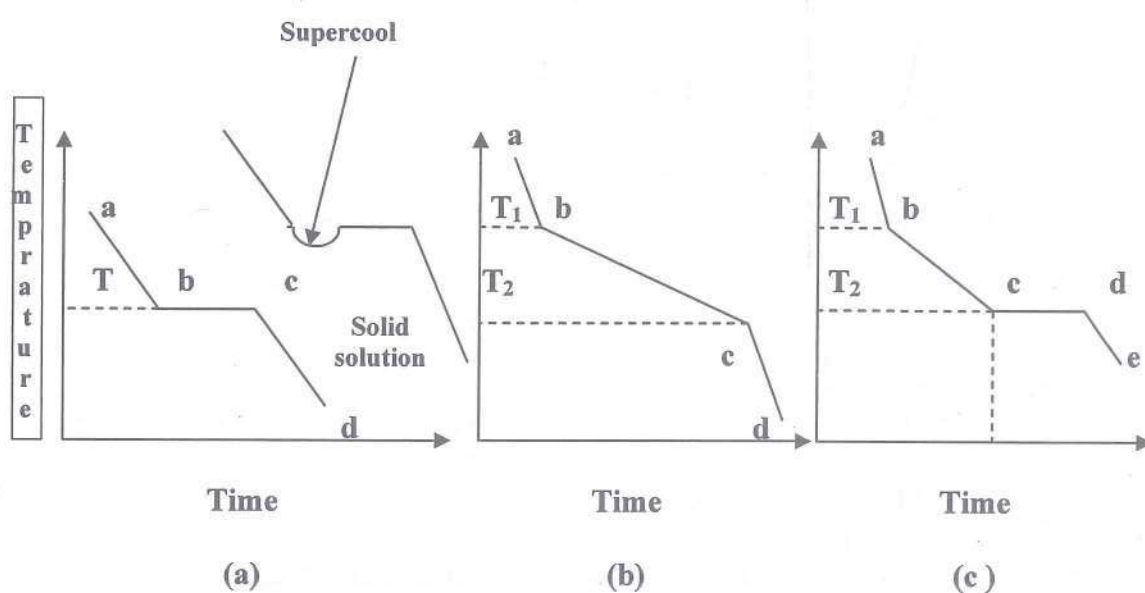


Figure 1. The effect of composition on the shape of cooling curves. (a)- Pure metal, (b)- two metals have been heated to point a where both are liquid and are dissolved in each other, (c)- two metals A and B are soluble in the liquid state but insoluble in the solid state

The arrival at the liquidus is usually clearly indicated by a marked reduction in the rate of cooling. Sometimes, in fact, there may be a small temporary rise of temperature at the start of freezing. This is an indication of undercooling, which must be minimized by slow cooling and continual stirring if accurate results are to be obtained. At certain compositions, e.g. pure metal, eutectic alloy, intermediate phases

which form directly from the melt, there is a clear arrest in the cooling curve when the latent heat of freezing is released at constant temperature. In most alloys however freezing occurs over a range of temperature, which produces a rounded cooling curve with a reduced slope. The final stage of freezing is usually rather poorly marked on the cooling curve; the solidus in this case is better determined by other methods, e.g. by heating curves or by quenching and microscopical examination. Where freezing is completed at a eutectic line, however, there is a sharp arrest when the eutectic part of the system freezes at constant temperature. The time of this arrest is proportional to the fraction of eutectic structure in the alloy and so, by plotting it against composition and extrapolating to zero time, the compositions of the solubility limits at the eutectic temperature can be found. Instead of plotting the time, directly against the temperature T , inverse rate curves are often constructed, in which dt/dT is plotted against T , as in Figure 2. These indicate the change points more sensitively. They can be constructed directly by plotting against temperature the times required for each successive unit drop in temperature.

In this experiment the cooling curves are used to construct the phase diagram. So, after constructing the cooling curves like those shown in Figure 1 for alloys of different compositions (from 0 % to 100% A), the projection of these curves to form a curve of temperature – compositions (phase diagram) should be done as shown in Figure 3.

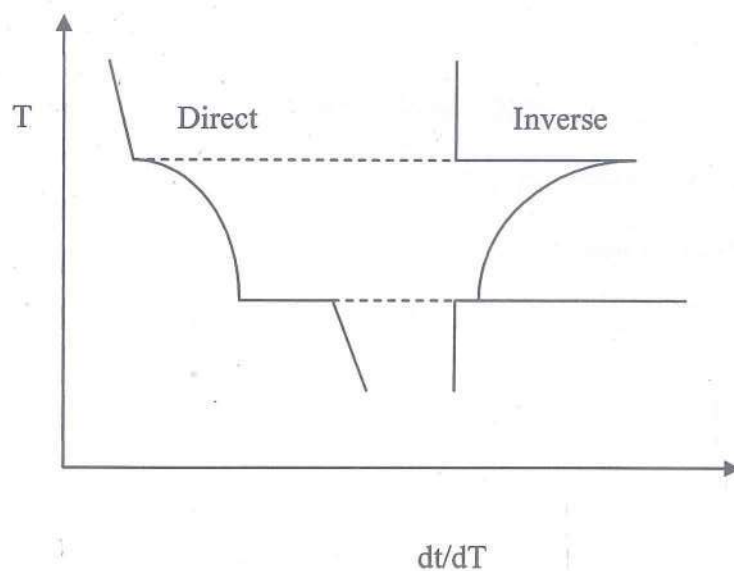


Figure 2. Direct and Inverse cooling curves

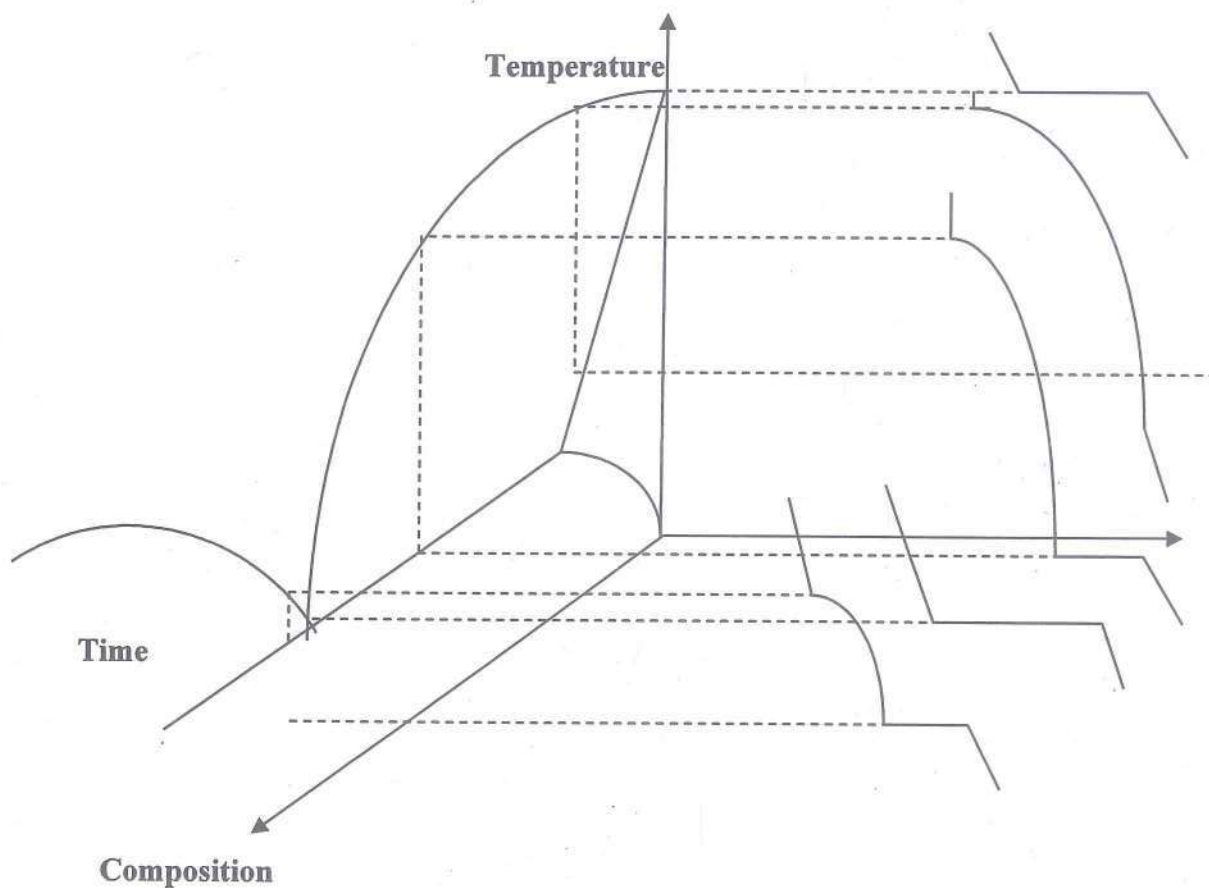


Figure 3. Projection of cooling curves (temperature – time coordinates) to form temperature composition curve (phase diagram)

IV. The Experiment:

Material

- ◆ Pure lead granules
- ◆ Pure Tin granules

Equipment

- ◆ Analytical balance
- ◆ Furnace
- ◆ Crucible, tongs, stand, thermometer; stopwatch

Procedure

- ◆ Weigh varying proportions of lead and Tin.
- ◆ Mix the two proportions in the crucible.
- ◆ Place the crucible into the furnace.
- ◆ Set the furnace temperature at 350C-400C
- ◆ Wait for the alloy to melt, then remove the crucible from the furnace and place it on the stand.
- ◆ Fix the thermometer.
- ◆ Record the temperature of the melt at intervals of 30 S.
- ◆ Continue stirring the melt until it solidifies.
- ◆ Plot a direct and inverse cooling curves, and indicate the arrest points.
- ◆ Construct the Pb-Sn phase diagram using the results of other groups.
- ◆ Groups of students investigating the Pb-Sn system should each prepare one of the alloys from the table below:

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
Pb	80%	60%	38%	20%
Sn	20%	40%	62%	80%

◆*Notes:*

i) Put the lead in the crucible provided.

ii) Melt the lead and then add the tin, stir gently and remove any scum formed.

Student Notes

Construction of equilibrium phase diagram

<i>Name</i>		<i>Section</i>	
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Results:

Cooling curve of tin – lead system

Tin-lead phase diagram.

Sectin V. Scientific terms and their meaning in Arabic

Arabic Translation of the scientific terms in this book.

<u>A</u>	
Absence	غياب
Accuracy	دقة ، اتقان ، احكام
Acoustic Emission Testing	الفحص بواسطة الانبعاث الصوتي
Agreement	موافقة
Apparatus	جهاز
Approach	سبيل ، مسلك ، منفذ
ASTM	الجمعية الامريكية للاختبار والمواد
<u>B</u>	
Band	حزمة ، عصبة
Bias	انحياز
Brinell Hardnes (HB)	الصلادة (القساوة) مقاسة بجهاز برنل
<u>C</u>	
Calibration	معايرة
Carburising	الكربنة
Cavity	تجويف ، نقرة
Chain	سلسلة
Characteristic	ميزة ، خاصة
Charcoal	فحم حيواني ا و نباتي
Chemical composition	التركيب الكيماوي
Chemical properties	الخصائص الكيميائية
Chemical solution	محلول كيماوي
Circumstances	حال ، واقع ، ظرف
Cleavage	شق ، انشقاق
Cold working	التشكيل على البارد
Complementary	متمم ، مكمل
Conclusion	ختام ، استنتاج ، نهاية
Condition	شرط
Correction	تصحيح
Cross section	مقطع عرضي
Crystal	بلورة
Curve	منحنى
<u>D</u>	
Dead zone	منطقة ساكنة ، منطقة موات
Defects	خلل ، علة ، عيب

Degree	درجة
Density	كثافة
Device	جهاز

Dial gauge	القرص المدرج
Dislocations	انخلاعات
Disturbance	اضطراب
Domain	مجال ، منطقة نفوذ
Drift	انحراف ، انحراف
Driving Force	القوة الدافعة (المحركة)

<u>E</u>	
Eddy Current Testing	الفحص بطريقة التيار الدوامي (الذي يخالف التيار الرئيسي)
Element	عنصر
Emery paper	ورق سنفرة
Environment	محيط ، وسط ، بيئة
Engineering strain	الانفعال الهندسي
Engineering stress	الاجهاد الهندسي
Equipment	معدات ، اجهزة
Error	خطأ
Estimation	تقدير
Etching	الحفر (الاظهار) بالحامض

<u>F</u>	
Facility	سهولة، تيسر، سبيل ميسر
Failure	اخذاق، فشل، انهيار
Fatigue	الكلال
Ferrous metals	معادن حديدية
Fidelity	أمانة (الاتصالات البعيدة)
File Test	الفحص بالمبرد
Flow line	خط التدفق، خط الانسياب

<u>G</u>	
Gap	فجوة
Grain	حببية
Grinding	صقل

<u>H</u>	
Hardness	قساوة
Heterogeneous	غير متجانس

Homogeneous	متجانس
Hot rolling	الدلفنة على الساخن
Hot shortness	قصيف (لا يقبل التشكيل) وهو ساخن
Hot working	التشكيل على الساخن
Human factors	العوامل الانسانية
Hypereutectic	ما بعد الانصهار الاصغري
Hypereutectoid	ما بعد اصغرائي الانصهار
Hypoeutectic	ما قبل الانصهار الاصغري
Hypo eutectoid	ما قبل اصغرائي الانصهار

I

Inclusion	محتسبات
Indenter	المحرز، الناقر
Indentation	نقر، علامة
Influence	تأثير
Instruments	اجهزة
Interpretation	تفسير
Interval	فترة
Intergranular	ما بين حبيبي

L

Laboratory	مختبر، مخبر
Lag	تخلف، تأخر
Lamella	رقائقي، صفائحي
Limit	حد، نهاية
Lower bound	الحد الاسفل
Liquidus	السيولة
Liquid Penetration Inspection	التفتيش بواسطة تخلخل السائل

M

Macroscopic Examination	الفحص الذي يمكن تحديده بالعين المجردة
Magnet Test	الفحص بالمغناطيس
Magnification	تكبير
Magnitude	كمية، مقدار
Matrix	مادة الترابط، القالب الام
Mean value	القيمة الوسطية (المتوسط)
Meaningful	ذو معنى
Measurement	قياس
Mechanical properties	خواص ميكانيكية

Metallography	ميتالوغرافيا: دراسة التركيب المعدني
Metallurgy	علم الفلزات
Microscope	المجهر
Microscopic examination	الفحص بواسطة المجهر
Microconstituent	مكون دقيق
Microvoids	فراغات دقيقة
Minimum	حد أدنى
Mounting	تلبيس

<u>N</u>	
Nitriding	النتردة
Nominal	اسمي، اعتباري
Nomenclature	تسمية، مجموعة مصطلحات
Non-ferrous	غير حديدي
Normal stress	الاجهاد الاسمي
Notation	تدليل رمزي - علامة
Nucleus	نواة

<u>O</u>	
Optical	ضوئي
Optical microscopy	دراسة التركيب المعدني بواسطة الضوء

<u>P</u>	
Parameter	مقدار متغير القيمة
Percentage	نسبة
Performance	اداء
Personnel	مستخدمون، اشخاص
Phase	طور
Phase diagram	المنحنى الطوري
Phenomena	ظاهرة
Physical properties	خواص فيزيقية
Plastic deformation	التشوه اللدن
Polishing	تلميع
Points	نقاط
Precipitation	ترسيب
Probability	احتمال
Proof stress	اجهاد الصمود

Probe

R

Radiography

التصوير بالأشعة

Random

عشوائي

Range

مدى، مجال

Recording

تسجيل، تدوين

Recrystallization

إعادة التبلور

Reproducibility

إمكانية الاستعادة

Replication

إعادة التجربة في ظروف مطابقة

Requirement

متطلب، شرط مقتضى

Resilience

رجوعية، ارتدادية الجسم المرن

Response

استجابة، تجاوب

S

Scale

مقياس مدرج، ميزان

Scratch test

الفحص بالخدش

Scattering

تبديد، بعثرة

Secant modulus

معامل القاطع

Sensitivity

حساسية

Segregation

الفصل التثاقلي (الفصل بالجاذبية)

Shop testing

الفحص في الورشة

Simultaneously perturbed

قلقلة متزامنة

Smooth

ناعم، أملس

Solidus

الجمود

Solid solution

محلول جامد

Solubility

الذوبانية، قابلية الذوبان

Span

امتداد، اتساع

Speed

سرعة

Stainless steel

فولاذ مقاوم للصدأ

Standard deviation

الانحراف المعياري

Static

ساكن

Statistical analysis

التحليل الإحصائي

Strain

انفعال

Strain hardening

الإصلاص الانفعالي

Strain rate

معدل الانفعال

Stress

اجهاد

Striation

حز، ثلم دقيق

System	منظومة
<u>T</u>	
Technical writing	الكتابة الفنية
T-distribution	التوزيع التائي
Temperature	حرارة
Tension	توتر
Theorem	نظرية
Tie line	خط توصيل
Toughness	صلابة، متانة
True strain	الانفعال الحقيقي
True stress	الاجهاد الحقيقي
<u>U</u>	
Ultimate tensile strength	اعلى مقاومة تحمل
Ultrasonic test	الفحص بالاهتزازات فوق السمعية
Unary phase diagram	المنحنى الطوري الاحادي
Uncertainty	ريبة، شك، الاحتمالية
Upper bound	الحد الاعلى
<u>V</u>	
Value	قيمة
Variable	متغير
Vickers hardness	المقاومة مقاسة بفكرز
Visual observation	الملاحظة العينية (البصرية)
<u>W</u>	
Wheel	عجل
Work hardening	القساوة بالتشكيل
<u>Y</u>	
Yield strength	مقاومة الخضوع
Young's modulus	معامل يونغ

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**Additional Useful Information as Appendices not cited in the
text of the book**

Appendix A:

Values of selected physical constants, unit abbreviations, SI multiple and submultiple prefixes, and unit conversion factors

A.1. Values of Selected Physical Constants

Quantity	Symbol	SI Units	egs Units
Avogadro's number	N_A	6.023×10^{23} molecules/mol	6.023×10^{23} molecules/mol
Boltzmann's constant erg/atom-K eV/atcm-K	k	1.38×10^{-23} I/atom-K	1.38×10^{-16} 8.62×10^{-5}
Bohr magneton erg/gauss	μ_B	9.27×10^{-24} A-rn ²	9.27×10^{-21}
Electron charge statcoulb	e	1.602×10^{-19} C	4.8×10^{-10}
Electron mass	—	9.11×10^{-31} kg	9.11×10^{-28} g
Gas constant	R	8.31 J/mol-K	1.987 cal/mol-K
Permeability of a vacuum	μ_0	1.257×10^{-6} henry/rn	unity ^a
Permittivity of a vacuum	ϵ_0	8.85×10^{-12} farad/rn	unity ^b
Planck's constant	h	6.63×10^{-34} J-s	6.63×10^{-27} erg-s 4.13×10^{-15} eV-s
Velocity of light in a vacuum	c	3×10^8 rn/s	3×10^{10} cm/s

A.2. Unit Abbreviations

A = ampere	in. = inch	N = Newton
Å = angstrom	J = joule	nm = nanometer
Btu = British thermal unit	K = degrees Kelvin	P = poise
C = Coulomb	kg = kilogram	Pa = pascal
°C = degrees Celsius	lb _f = pound force	s = second
cal = calorie (gram)	lbm = pound mass	T = temperature
cm = centimeter	rn = meter	μrn = micrometer
eV = electron volt	Mg = megagram	(micron)
°F = degrees Fahrenheit	mm = millimeter	W = watt
ft = foot	mol = mole	psi = pounds per square
inch	MPa = megapascal	inch
g = gram		

A.3. SI Multiple and Submultiple Prefixes

<i>Factor by Which Multiplied</i>	<i>Prefix</i>	<i>Symbol</i>
10^9	giga	G
10^6	mega	M
10^3	kilo	k
10^{-2}	centi ^a	c
10^{-3}	milli	m
10^{-6}	micro	μ
10^{-9}	nano	n
10^{-12}	pico	p

A.4. Unit conversion factors

Length

$1 \text{ m} = 10^{10} \text{ A}^\circ$	$1 \text{ A}^\circ = 10^{-10} \text{ m}$
$1 \text{ m} = 10^9 \text{ nm}$	$1 \text{ nm} = 10^{-9} \text{ m}$
$1 \text{ m} = 10^6 \text{ }\mu\text{m}$	$1 \text{ }\mu\text{m} = 10^{-6} \text{ m}$
$1 \text{ m} = 10^3 \text{ mm}$	$1 \text{ mm} = 10^{-3} \text{ m}$
$1 \text{ m} = 10^2 \text{ cm}$	$1 \text{ cm} = 10^{-2} \text{ m}$
$1 \text{ mm} = 0.0394 \text{ in.}$	$1 \text{ in.} = 25.4 \text{ mm}$
$1 \text{ cm} = 0.394 \text{ in.}$	$1 \text{ in.} = 2.54 \text{ cm}$
$1 \text{ m} = 3.28 \text{ ft}$	$1 \text{ ft} = 0.3048 \text{ m}$

Area

$1 \text{ m}^2 = 10^4 \text{ cm}^2$	$1 \text{ cm}^2 = 10^{-4} \text{ m}^2$
$1 \text{ mm}^2 = 10^{-2} \text{ cm}^2$	$1 \text{ cm}^2 = 10^2 \text{ mm}^2$
$1 \text{ m}^2 = 10.76 \text{ ft}^2$	$1 \text{ ft}^2 = 0.093 \text{ m}^2$
$1 \text{ cm}^2 = 0.1550 \text{ in}^2$	$1 \text{ in}^2 = 6.452 \text{ cm}^2$

Volume

$1 \text{ m}^3 = 10^6 \text{ cm}^3$	$1 \text{ cm}^3 = 10^{-6} \text{ m}^3$
$1 \text{ mm}^3 = 10^{-3} \text{ cm}^3$	$1 \text{ cm}^3 = 10^3 \text{ mm}^3$
$1 \text{ m}^3 = 35.32 \text{ ft}^3$	$1 \text{ ft}^3 = 0.0283 \text{ m}^3$
$1 \text{ cm}^3 = 0.0610 \text{ in}^3$	$1 \text{ in}^3 = 16.39 \text{ cm}^3$

Mass

$1 \text{ Mg} = 10^3 \text{ kg}$	$1 \text{ kg} = 10^{-3} \text{ Mg}$
$1 \text{ kg} = 10^3 \text{ g}$	$1 \text{ g} = 10^{-3} \text{ kg}$
$1 \text{ kg} = 2.205 \text{ lb}_m$	$1 \text{ lb}_m = 0.4536 \text{ kg}$
$1 \text{ kg} = 2.205 \times 10^{-3} \text{ lb}_m$	$1 \text{ lb}_m = 453.6 \text{ g}$

Density

$$\begin{aligned}1 \text{ kg/m}^3 &= 10^{-3} \text{ g/cm}^3 \\1 \text{ Mg/m}^3 &= 1 \text{ g/cm}^3 \\1 \text{ kg/m}^3 &= 0.0624 \text{ lb}_m/\text{ft}^3 \\1 \text{ g/cm}^3 &= 62.4 \text{ lb}_m/\text{ft}^3 \\1 \text{ g/cm}^3 &= 0.0361 \text{ lb}_m/\text{in.}^3\end{aligned}$$

$$\begin{aligned}1 \text{ g/cm}^3 &= 10^3 \text{ kg/m}^3 \\1 \text{ g/cm}^3 &= 1 \text{ Mg/m}^3 \\1 \text{ lb}_m/\text{ft}^3 &= 16.02 \text{ kg/m}^3 \\1 \text{ lb}_m/\text{ft}^3 &= 1.602 \times 10^{-2} \text{ g/cm}^3 \\1 \text{ lb}_m/\text{in.}^3 &= 27.7 \text{ g/cm}^3\end{aligned}$$

Force

$$\begin{aligned}1 \text{ N} &= 10^5 \text{ dynes} \\1 \text{ N} &= 0.2248 \text{ lb}_f\end{aligned}$$

$$\begin{aligned}1 \text{ dyne} &= 10^{-5} \text{ N} \\1 \text{ lb}_f &= 4.448 \text{ N}\end{aligned}$$

Stress

$$\begin{aligned}1 \text{ MPa} &= 145 \text{ psi} \\1 \text{ MPa} &= 0.102 \text{ kg/mm}^2 \\1 \text{ Pa} &= 10 \text{ dynes/cm}^2 \\1 \text{ kg/mm}^2 &= 1422 \text{ psi}\end{aligned}$$

$$\begin{aligned}1 \text{ psi} &= 6.90 \times 10^{-3} \text{ MPa} \\1 \text{ kg/mm}^2 &= 9.806 \text{ MPa} \\1 \text{ dyne/cm}^2 &= 0.10 \text{ Pa} \\1 \text{ psi} &= 7.03 \times 10^{-4} \text{ kg/mm}^2\end{aligned}$$

Fracture Toughness

$$1 \text{ psi in.}^{1/2} = 1.099 \times 10^{-3} \text{ MPa m}^{1/2} \quad 1 \text{ MPa m}^{1/2} = 910 \text{ psi in.}^{1/2}$$

Energy

$$\begin{aligned}1 \text{ J} &= 10^7 \text{ ergs} \\1 \text{ J} &= 6.24 \times 10^{18} \text{ eV} \\1 \text{ J} &= 0.239 \text{ cal} \\1 \text{ J} &= 9.48 \times 10^{-4} \text{ Btu} \\1 \text{ J} &= 0.738 \text{ ft-lb}_f \\1 \text{ eV} &= 3.83 \times 10^{-20} \text{ cal} \\1 \text{ cal} &= 3.97 \times 10^{-3} \text{ Btu}\end{aligned}$$

$$\begin{aligned}1 \text{ erg} &= 10^{-7} \text{ J} \\1 \text{ eV} &= 1.602 \times 10^{-19} \text{ J} \\1 \text{ cal} &= 4.184 \text{ J} \\1 \text{ btu} &= 1054 \text{ J} \\1 \text{ ft-lb}_f &= 1.356 \text{ J} \\1 \text{ cal} &= 2.61 \times 10^{19} \text{ eV} \\1 \text{ Btu} &= 252.0 \text{ cal}\end{aligned}$$

Power

$$\begin{aligned}1 \text{ W} &= 0.239 \text{ cal/s} \\1 \text{ W} &= 3.414 \text{ Btu/h} \\1 \text{ cal/s} &= 14.29 \text{ Btu/h}\end{aligned}$$

$$\begin{aligned}1 \text{ cal/s} &= 4.184 \text{ W} \\1 \text{ Btu/h} &= 0.293 \text{ W} \\1 \text{ Btu/h} &= 0.070 \text{ cal/s}\end{aligned}$$

Viscosity

$$1 \text{ Pa-s} = 10 \text{ P}$$

$$1 \text{ P} = 0.1 \text{ Pa-s}$$

Temperature, T

$$\begin{aligned}T(\text{K}) &= 273 + T(^{\circ}\text{C}) \\T(\text{K}) &= 5/9 [T(^{\circ}\text{F}) - 32] + 273 \\T(^{\circ}\text{C}) &= 5/9 [T(^{\circ}\text{F}) - 32]\end{aligned}$$

$$\begin{aligned}T(^{\circ}\text{C}) &= T(\text{K}) - 273 \\T(^{\circ}\text{F}) &= 9/5 [T(\text{K}) - 273] + 32 \\T(^{\circ}\text{F}) &= 9/5 [T(^{\circ}\text{C})] + 32\end{aligned}$$

Specific Heat

$$\begin{aligned}1 \text{ J/kg-K} &= 2.39 \times 10^{-4} \text{ cal/g-K} \\1 \text{ J/kg-K} &= 2.39 \times 10^{-4} \text{ Btu/lbm-}^{\circ}\text{F} \\1 \text{ cal/g-}^{\circ}\text{C} &= 1.0 \text{ Btu/lbm-}^{\circ}\text{F}\end{aligned}$$

$$\begin{aligned}1 \text{ cal/g-}^{\circ}\text{C} &= 4184 \text{ J/kg-K} \\1 \text{ Btu/lbm-}^{\circ}\text{F} &= 4184 \text{ J/kg-K} \\1 \text{ Btu/lbm-}^{\circ}\text{F} &= 1.0 \text{ cal/g-K}\end{aligned}$$

Thermal Conductivity

$$\begin{aligned}1 \text{ W/m-K} &= 2.39 \times 10^{-3} \text{ cal/cm-s-K} \\1 \text{ W/m-K} &= 0.578 \text{ Btu/ft-h-}^{\circ}\text{F} \\1 \text{ cal/cm-s-K} &= 241.8 \text{ Btu/ft-h-}^{\circ}\text{F}\end{aligned}$$

$$\begin{aligned}1 \text{ cal/cm-s-K} &= 418.4 \text{ W/m-K} \\1 \text{ Btu/ft-h-}^{\circ}\text{F} &= 1.730 \text{ W/m-}^{\circ}\text{F} \\1 \text{ Btu/ft-h-}^{\circ}\text{F} &= 4.136 \times 10^{-3} \text{ cal/cm-s-K}\end{aligned}$$

Appendix B:

**Physical and chemical data for selected elements, Atomic and ionic radii of
selected elements.**

B.1. Physical and Chemical Data for Selected Elements

<u>Number</u>	<u>Element</u>	<u>Symbol</u>	<u>Atomic Mass (amu)</u>	<u>Density of Solid at 20 °C (Mg/in³ = g/cm³)</u>	<u>Crystal Structure (at 20°C)</u>	<u>Melting Point (°C)</u>	<u>Atomic Number</u>
I	Hydrogen	H	1.008			—259.14	
2	Helium	He	4.003			—272.2	2
3	Lithium	Li	6.941	0.533	bcc	180.54	3
4	Beryllium	Be	9.012	1.85	hcp	1278	4
5	Boron	B	10.81	2.47		2300	5
6	Carbon	C	12.01	2.27	hex.	—3550	6
7	Nitrogen	N	14.01			—209.86	7
8	Oxygen	O	16.00			—218.4	8
9	Fluorine	F	19.00			—219.62	9
10	Neon	Ne	20.8			—248.67	10
11	Sodium	Na	22.99	0.966	bcc	97.81	11
12	Magnesium	Mg	24.31	.74	hcp	648.8	12
13	Aluminum	Al	26.98	2.70	fcc	660.37	13
14	Silicon	Si	28.09	2.33	dia. cub.	1410	14
15	Phosphorus	P	30.97	1.82 (white)	ortho.	44.1 (white)	15
16	Sulfur	S	32.06	2.09	ortho.	112.8	16
17	Chlorine	Cl	35.45			—100.98	17
18	Argon	Ar	39.95			189.2	18
19	Potassium	K	39.10	0.862	bcc	63.65	19
20	Calcium	Ca	40.08	1.53	fcc	839	20
21	Scandium	Sc	44.96	2.99	fcc	1539	21
22	Titanium	Ti	47.90	4.51	hcp	1660	22
23	Vanadium	V	50.94	6.09	bcc	1890	23
24	Chromium	Cr	52.00	7.19	bcc	1857	24
25	Manganese	Mn	54.94	7.47	cubic	1244	25
26	Iron	Fe	55.85	7.87	bcc	1535	26
27	Cobalt	Co	58.93	8.8	hcp	1495	27
28	Nickel	Ni	58.69	8.91	fcc	1453	28
29	Copper	Cu	63.55	8.93	fcc	1083.4	29
30	Zinc	Zn	65.38	7.13	hcp	419.58	30
31	Gallium	Ga	69.72	5.91	ortho.	2978	31
32	Germanium	Ge	72.59	5.32	dia. cub.	937>4	32
33	Arsenic	As	74.92	5.78	rhomb.	817 (28 atm)	33
34	Selenium	Se	78.96	4.81	hex~	217	34
35	Bromine	Br	79.90			—7.2	35
36	Krypton	Kr	83.80			—156.6	36
37	Rubidium	Rb	85.47	1.53	bcc	38.89	37
38	Strontium	Sr	87.62	2.58	fcc	769	38
39	Yttrium	Y	88.91	4.48	hcp	1523	39
40	Zirconium	Zr	91.22	6.51	hcp	1852	40
41	Niobium	Nb	92.91	8.58	bcc	2468	41
42	Molybdenum	Mo	95.94	10.22	bcc	2617	42

Continued

Atomic		Atomic		Density	Crystal		
Mass		Mass		of Solid	Structure		
				at 20 °C	(at 20°C)		
					Melting		
Atomic							
Number	Element	Symbol	(amu)	(Mg/in ³ = g/cm ³)	Point	(°C)	Number
43	Technetium	Tc	98.91	11.50	hcp	2172	43
44	Ruthenium	Ru	101.07	12.36	hcp	2310	44
45	Rhodium	Rh	102.91	12.42	fcc	1966	45
46	Palladium	Pd	106.4	12.00	fcc	1552	46
47	Silver	Ag	107.87	10.50	fcc	961.93	47
48	Cadmium	Cd	112.4	8.65	hcp	320.9	48
49	Indium	In	114.82	7.29	fct	156.61	49
50	Tin	Sn	118.69	7.29	bct	231.9681	50
SI	Antimony	Sb	121.75	6.69	rhomb.	630.74	SI
52	Tellurium	Te	127.60	6.25	hex.	449.5	52
53	Iodine	I	126.90	4.95	ortho.	113.5	53
54	Xenon	Xe	131.30			—111.9	54
55	Cesium	Cs	132.91	1.91	bcc	28.40	55
				(~100)			
56	Barium	Ba	137.33	3.59	bcc	725	56
57	Lanthanum	La	138.91	6.17	hex.	920	57
58	Cerium	Ce	140.12	6.77	fcc	798	58
59	Praseodymium	Pr	140.91	6.78	hex.	931	59
60	Neodymium	Nd	144.24	7.00	hex.	1010	60
61	Promethium	Pm	(145)		hex.	—1080	61
62	Samarium	Sm	150.4	7.54	rhomb.	1072	62
63	Europium	Eu	151.96	5.25	bcc	822	63
64	Gadolinium	Gd	157.25	7.87	hcp	1311	64
65	Terbium	Tb	158.93	8.27	hcp	1360	65
66	Dysprosium	Dy	162.50	8.53	hcp	1409	66
67	Holmium	Ho	164.93	8.80	hcp	1470	67
68	Erbium	Er	167.26	9.04	hcp	1522	68
69	Thulium	Tm	168.93	9.33	hcp	1545	69
70	Ytterbium	Yb	173.04	6.97	fcc	824	30
71	Lutetium	Lu	174.97	9.84	hcp	1656	71
72	Hafnium	Hf	178.49	13.28	hcp	2227	72
73	Tantalum	Ta	180.95	16.67	bcc	2996	73
74	Tungsten	W	183.85	19.25	bcc	3410	74
75	Rhenium	Re	186.2	21.02	hcp	3180	75
76	Osmium	Os	190.2	22.58	hcp	3045	76
77	Iridium	Ir	192.22	22.55	fcc	2410	77
78	Platinum	Pt	195.09	21.44	fcc	1772	78
79	Gold	Au	96.97	19.28	fcc	1064.43	79
80	Mercury	Hg	200.59			—38.87	80
SI	Thallium	Tl	204.37	11.87	hcp	303.5	81
82	Lead	Pb	207.2	11.34	fcc	327.502	82
83	Bismuth	Bi	208.98	9.80	rhomb.	271.3	83
92	Uranium	U	238.03	19.05	ortho.	1132	92

B.2. Atomic and Ionic Radii of Selected Elements

Atomic Number	Symbol	Atomic Radius (nm)	Ion with Most Common Valence	Ionic Radius (nm)
3	Li	0.152	Li ⁺	0.078
4	Be	0.114	Be ²⁺	0.054
5	B	0.097	B ³⁺	0.02
6	C	0.077	C ⁴⁺	<0.02
7	N	0.071	N ⁵⁺	0.01—0.02
8	O	0.060	O ²⁻	0.132
9	F	—	F ⁻	0.133
11	Na	0.186	Na ⁺	0.098
12	Mg	0.160	Mg ²⁺	0.078
13	Al	0.143	Al ³⁺	0.057
14	Si	0.117	Si ⁴⁺	0.039
15	P	0.109	P ⁵⁺	0.03—0.04
16	S	0.106	S ²⁻	0.174
17	Cl	0.107	Cl ⁻	0.181
19	K	0.231	K ⁺	0.133
20	Ca	0.197	Ca ²⁺	0.106
21	Sc	0.160	Sc ³⁺	0.083
22	Ti	0.147	Ti ⁴⁺	0.064
23	V	0.132		0.061
24	Cr	0.125	Cr ³⁺	0.064
25	Mn	0.112	Mn ²⁺	0.091
26	Fe	0.124	Fe ²⁺	0.087
27	Co	0.125	Co ²⁺	0.082
28	Ni	0.125	Ni ²⁺	0.078
29	Cu	0.128	Cu ⁺	0.096
30	Zn	0.133	Zn ²⁺	0.083
31	Ga	0.135	Ga ³⁺	0.062
32	Ge	0.122	Ge ⁴⁺	0.044
35	Br	0.119	Br ⁻	0.196
39	Y	0.181		0.106
40	Zr	0.158	Zr ⁴⁺	0.087
41	Nb	0.143	Nb ⁵⁺	0.074
42	Mo	0.136	Mo ⁶⁺	0.068
46	Pd	0.137	Pd ²⁺	0.050
47	Ag	0.144	Ag ⁺	0.113
48	Cd	0.150	Cd ²⁺	0.103
50	Sn	0.158	Sn ⁴⁺	0.074
53	I	0.136	I ⁻	0.220
55	Cs	0.265	Cs ⁺	0.165
56	Ba	0.217	Ba ²⁺	0.143
74	W	0.137		0.068
78	Pt	0.138	Pt ²⁺	0.052
79	Au	0.144	Au ⁺	0.137
80	Hg	0.150	Hg ²⁺	0.112
82	Pb	0.175	Pb ²⁺	0.132
92	U	0.138	U ⁴⁺	0.105

Appendix C:

Diffusion data for selected materials

<u>Diffusion Couple</u>	<u>Activation energy (Q)</u> (cal/mol)	<u>D₀</u> (cm ² /s)
<u>Interstitial diffusion:</u>		
C in FCC iron	32,900	0.23
C in BCC iron	20,900	0.011
N in FCC iron	34,600	0.0034
N in BCC iron	18,300	0.0047
H in FCC iron	10,300	0.0063
H in BCC iron	3,600	0.0012

Self-diffusion (vacancy diffusion):

Pb in FCC Pb	25,900	1.27
Al in FCC Al	32,200	0.10
Cu in FCC Cu	49,300	0.36
Fe in FCC Fe	66,700	0.65
Zn in HCP Zn	21,800	0.1
Mg in HCP Mg	32,200	1.0
Fe in BCC Fe	58,900	4.1
W in BCC W	143,300	1.88
Si in Si (covalent)	110,000	1800.0
C in C (covalent)	163,000	5.0

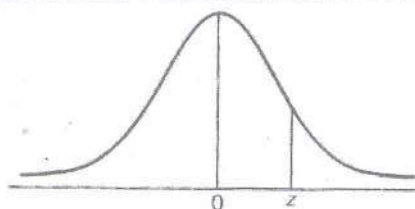
Heterogeneous diffusion (vacancy diffusion):

Ni in Cu	57,900	2.3
Cu in Ni	61,500	0.65
Zn in Cu	43,900	0.78
Ni in FCC iron	64,000	4.1
Au in Ag	45,500	0.26
Ag in Au	40,200	0.072
Al in Cu	39,500	0.045
Al in Al₂O₃	114,000	28.0
O in Al₂O₃	152,000	1900.0
Mg in MgO	79,000	0.249
O in MgO	82,100	0.000043

From several sources including Y. Adda and S. Philiberi, *La Diffusion dans les Solides*. Vol.2. 1966.

Appendix D normal and t-distributions tables D.1. Normal distribution tables

Areas under the standard normal curve

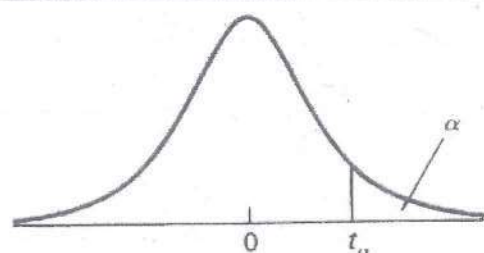


Second Decimal Place in z										
z	0.00	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
0.0	0.0000	0.0040	0.0080	0.0120	0.0160	0.0199	0.0239	0.0279	0.0319	0.0359
0.1	0.0398	0.0438	0.0478	0.0517	0.0557	0.0596	0.0636	0.0675	0.0714	0.0753
0.2	0.0793	0.0832	0.0871	0.0910	0.0948	0.0987	0.1026	0.1064	0.1103	0.1141
0.3	0.1179	0.1217	0.1255	0.1293	0.1331	0.1368	0.1406	0.1443	0.1480	0.1517
0.4	0.1554	0.1591	0.1628	0.1664	0.1700	0.1736	0.1772	0.1808	0.1844	0.1879
0.5	0.1915	0.1950	0.1985	0.2019	0.2054	0.2088	0.2123	0.2157	0.2190	0.2224
0.6	0.2257	0.2291	0.2324	0.2357	0.2389	0.2422	0.2454	0.2486	0.2517	0.2549
0.7	0.2580	0.2611	0.2642	0.2673	0.2704	0.2734	0.2764	0.2794	0.2823	0.2852
0.8	0.2881	0.2910	0.2939	0.2967	0.2995	0.3023	0.3051	0.3078	0.3106	0.3133
0.9	0.3159	0.3186	0.3212	0.3238	0.3264	0.3289	0.3315	0.3340	0.3365	0.3389
1.0	0.3413	0.3438	0.3461	0.3485	0.3508	0.3531	0.3554	0.3577	0.3599	0.3621
1.1	0.3643	0.3665	0.3686	0.3708	0.3729	0.3749	0.3770	0.3790	0.3810	0.3830
1.2	0.3849	0.3869	0.3888	0.3907	0.3925	0.3944	0.3962	0.3980	0.3997	0.4015
1.3	0.4032	0.4049	0.4066	0.4082	0.4099	0.4115	0.4131	0.4147	0.4162	0.4177
1.4	0.4192	0.4207	0.4222	0.4236	0.4251	0.4265	0.4279	0.4292	0.4306	0.4319
1.5	0.4332	0.4345	0.4357	0.4370	0.4382	0.4394	0.4406	0.4418	0.4429	0.4441
1.6	0.4452	0.4463	0.4474	0.4484	0.4495	0.4505	0.4515	0.4525	0.4535	0.4545
1.7	0.4554	0.4564	0.4573	0.4582	0.4591	0.4599	0.4608	0.4616	0.4625	0.4633
1.8	0.4641	0.4649	0.4656	0.4664	0.4671	0.4678	0.4686	0.4693	0.4699	0.4706
1.9	0.4713	0.4719	0.4726	0.4732	0.4738	0.4744	0.4750	0.4756	0.4761	0.4767
2.0	0.4772	0.4778	0.4783	0.4788	0.4793	0.4798	0.4803	0.4808	0.4812	0.4817
2.1	0.4821	0.4826	0.4830	0.4834	0.4838	0.4842	0.4846	0.4850	0.4854	0.4857
2.2	0.4861	0.4864	0.4868	0.4871	0.4875	0.4878	0.4881	0.4884	0.4887	0.4890
2.3	0.4893	0.4896	0.4898	0.4901	0.4904	0.4906	0.4909	0.4911	0.4913	0.4916
2.4	0.4918	0.4920	0.4922	0.4925	0.4927	0.4929	0.4931	0.4932	0.4934	0.4936
2.5	0.4938	0.4940	0.4941	0.4943	0.4945	0.4946	0.4948	0.4949	0.4951	0.4952
2.6	0.4953	0.4955	0.4956	0.4957	0.4959	0.4960	0.4961	0.4962	0.4963	0.4964
2.7	0.4965	0.4966	0.4967	0.4968	0.4969	0.4970	0.4971	0.4972	0.4973	0.4974
2.8	0.4974	0.4975	0.4976	0.4977	0.4977	0.4978	0.4979	0.4979	0.4980	0.4981
2.9	0.4981	0.4982	0.4982	0.4983	0.4984	0.4984	0.4985	0.4985	0.4986	0.4986
3.0	0.4987	0.4987	0.4987	0.4988	0.4988	0.4989	0.4989	0.4989	0.4990	0.4990
3.1	0.4990	0.4991	0.4991	0.4991	0.4992	0.4992	0.4992	0.4992	0.4993	0.4993
3.2	0.4993	0.4993	0.4994	0.4994	0.4994	0.4994	0.4994	0.4995	0.4995	0.4995
3.3	0.4995	0.4995	0.4995	0.4996	0.4996	0.4996	0.4996	0.4996	0.4996	0.4997
3.4	0.4997	0.4997	0.4997	0.4997	0.4997	0.4997	0.4997	0.4997	0.4997	0.4998
3.5	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998	0.4998
3.6	0.4998	0.4998	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
3.7	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
3.8	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999	0.4999
3.9	0.5000*									

* For $z \geq 3.90$, the areas are 0.5000 to four decimal places.

D.2.t-distribution table

Student's t -distribution (values of $t_{\alpha, v}$)



v	$t_{0.10}$	$t_{0.05}$	$t_{0.025}$	$t_{0.01}$	$t_{0.005}$	v
1	3.078	6.314	12.706	31.821	63.657	1
2	1.886	2.920	4.303	6.965	9.925	2
3	1.638	2.353	3.182	4.541	5.841	3
4	1.533	2.132	2.776	3.747	4.604	4
5	1.476	2.015	2.571	3.365	4.032	5
6	1.440	1.943	2.447	3.143	3.707	6
7	1.415	1.895	2.365	2.998	3.499	7
8	1.397	1.860	2.306	2.896	3.355	8
9	1.383	1.833	2.262	2.821	3.250	9
10	1.372	1.812	2.228	2.764	3.169	10
11	1.363	1.796	2.201	2.718	3.106	11
12	1.356	1.782	2.179	2.681	3.055	12
13	1.350	1.771	2.160	2.650	3.012	13
14	1.345	1.761	2.145	2.624	2.977	14
15	1.341	1.753	2.131	2.602	2.947	15
16	1.337	1.746	2.120	2.583	2.921	16
17	1.333	1.740	2.110	2.567	2.898	17
18	1.330	1.734	2.101	2.552	2.878	18
19	1.328	1.729	2.093	2.539	2.861	19
20	1.325	1.725	2.086	2.528	2.845	20
21	1.323	1.721	2.080	2.518	2.831	21
22	1.321	1.717	2.074	2.508	2.819	22
23	1.319	1.714	2.069	2.500	2.807	23
24	1.318	1.711	2.064	2.492	2.797	24
25	1.316	1.708	2.060	2.485	2.787	25
26	1.315	1.706	2.056	2.479	2.779	26
27	1.314	1.703	2.052	2.473	2.771	27
28	1.313	1.701	2.048	2.467	2.763	28
29	1.311	1.699	2.045	2.462	2.756	29
∞	1.282	1.645	1.960	2.326	2.576	∞

